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PROJECT PLAN

for the

VASQUEZ BOULEVARD AND I-70 RESIDENTIAL RISK-BASED SAMPLING

STAGE I INVESTIGATION

DENVER, CO

August 1998

Prepared For:
U.S. Environmental Protection Agency, Region VIII
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A1 APPROVAL PAGE

This Project Plan for the Vasquez Boulevard and I-70 Residential Risk-based Sampling Stage I Investigation has been prepared at the request of the U.S. Environmental Agency, Region 8, by ISSI, Inc. Study investigations and activities addressed in this Project Plan are approved without conditions.

Program Approval — 8/10/98 date
Bonita Lavelle
EPA Remedial Project Manager
Vasquez Boulevard and I-70 Site

for Program Approval — 8/10/98 date
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EPA On Scene Coordinator
Vasquez Boulevard and I-70 Site

Technical Approval — 7/31/98 date
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EPA Regional Toxicologist
Office of Ecosystems Protection and Remediation

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A3 DISTRIBUTION LIST

This Draft Vasquez Boulevard and I-70 Residential Risk-based Sampling - Stage I Investigation Project Plan and any revisions will be distributed as follows:

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A4 PROJECT TASK AND ORGANIZATION

A4.1 Project Task

EPA Region VIII is working in cooperation with the City and County of Denver and the Colorado Department of Public Health and Environment (CDPHE) to further characterize surface soils and other environmental and biologic media at selected residences in the Vasquez Boulevard and I-70 Site. This document serves as the Sampling and Analysis Plan and Quality Assurance Project Plan (SAP/QAPP) for the project and presents the organization, objectives, functional activities and specific quality assurance and quality control activities associated with this investigation. This SAP/QAPP includes site background information, project objectives and scope, sampling design and rationale, analytical design and rationale and data quality objectives (DQOs) to support these activities. It describes the specific protocols that will be followed for sampling, processing of samples, storage, chain of custody, and laboratory analyses.

A4.2 Project Organization

The following lists key personnel who will serve as contacts and provide technical expertise during implementation of this Project Plan.

U.S. Environmental Protection Agency

- Bonita Lavelle, EPA Remedial Project Manager, will be responsible for overall project management, technical oversight and coordination among EPA and its contractors, the State of Colorado and the City and County of Denver. Ms. Lavelle will be a principal decision-maker for this project.

- Peter Stevenson, EPA On-Scene Coordinator, will be responsible for implementing and documenting some of the field sampling activities in accordance with this SAP/QAPP.

- Christopher P. Weis, Ph.D., EPA Regional Toxicologist, will serve as the primary technical contact for this project. He will be responsible for evaluating the human health risk to residents of Vasquez Boulevard and I-70 site. Dr. Weis will be a principal data user and decision-maker for this project.

- William Brattin, Ph.D., ISSI, Inc., will be responsible for managing ISSI's activities which include: preparation of planning documents, providing technical oversight, and compiling and summarizing data generated during the supplemental investigations. Dr. Brattin will be a principal data user for this project.

A5 PROBLEM DEFINITION AND BACKGROUND

A5.1 Background

In the Spring of 1998, a large-scale surface soil sampling program was implemented to determine the nature and extent of heavy metal contamination within the Vasquez Boulevard and I-70 Site. Note that boundaries have not yet been delineated for this site. The Spring 1998 investigation, carried out on behalf of the USEPA Region 8 by URS Operating Services (UOS), measured bulk concentrations of arsenic, cadmium and lead in approximately 2400 surface soil samples. Each of the nearly 2400 surface soil sample locations were collected individually (grab sample), typically with one sample from the front yard and one sample from the back yard of each residential property. The raw surface soil was homogenized and sieved to particles less than 2 mm ("bulk" soil) prior to concentration measurement. The range of concentrations for these surface soils were:

Analyte	Concentration Range (ppm)
Arsenic	< 44 – 5600
Cadmium[a]	< 96 – 120
Lead	< 28 – 8000

a – Although concentration ranges are available within the UOS Surface Soil Database, these data were determined unusable and were, therefore not reported in the UOS final report (USEPA 1998b).

As a result of the comprehensive surface soil investigation performed by UOS in Spring 1998, approximately 37 residences were identified as having surface soil concentrations in the front and/or back yard above the removal action levels of 400 ppm for arsenic and 2,000 ppm for lead. These action levels have been defined to identify residences that may require immediate soil removal.

A6 PROJECT TASK DESCRIPTION

This section outlines the overall study goals and study objectives of the Vasquez Boulevard and I-70 Residential Risk-based Sampling - Stage I Investigation. The study goals outline the unique endpoints that are desired at the completion of the project. The study objectives identify the steps required to attain each goal.

A6.1 Study Goals

STUDY GOALS: EPA has three distinct goals for this study as outlined below:

1) Characterize the nature and extent of arsenic (As), cadmium (Cd), lead (Pb) and zinc (Zn) contamination within selected residential yards by performing high-density ("intensive") sampling of surface soil and soil cores. Characterize a fraction of the surface and core soils for an extensive list of metals (Section B4.2.1) to evaluate potential contaminants of concern (PCOCs).

2) Quantify the concentrations of arsenic, cadmium, lead and zinc in the following environmental media at residences identified for soil removal action:

   - indoor household dust (As, Cd, Pb, Zn)
   - attic dust (As, Cd, Pb, Zn)
   - tap water (Pb only)
   - exterior and interior paint (Pb only)
   - garden vegetables (As, Cd, Pb, Zn)
   - surface soil samples co-located with garden vegetables (As, Cd, Pb, Zn)

3) Estimate the extent to which residents at properties identified for soil removal action are presently exposed to arsenic and lead by performing a voluntary human biomonitoring program to quantify levels of these metals in biological media. Three parameters will be measured as part of the biomonitoring program:

   - Arsenic in composite hair samples
   - Inorganic arsenic in first void urine samples
   - Blood lead

It is envisioned that this investigation will proceed in two distinct phases. The first phase will implement the objectives for Study Goal #1 (Intensive Surface Soil and Core Sampling). The second phase (Environmental Sampling and Biomonitoring at Selected Residences) will proceed after residences requiring soil removal action are identified.

A6.2 Study Objectives

This project consists of several steps to define the magnitude of possible metal exposures to residents at the Vasquez Boulevard and I-70 Site. The objectives for each study goal and their intended use are outlined in subsequent paragraphs.

STUDY GOAL #1

Study Objective #1-1: Choose the 5 residences within the study area having the greatest reported surface soil arsenic concentration in the Spring 1998 sampling program (USEPA 1998b). Collect surface soil samples on a 5'x5' grid to characterize the spatial relationship of metals (As, Cd, Pb, Zn) concentrations at each property. Whenever possible, this grid sampling program will extend out approximately 15 feet (3 grid nodes) to properties adjacent to the targeted residences where yards are contiguous. This information will be used to:

1) Determine the mean metals concentrations for the front and back yards at each residence. Evaluate whether metals concentrations differ significantly between front and back yards.

2) Evaluate the spatial distribution of arsenic and lead concentrations at the target residence in order to judge whether the grab sample concentrations for each residence (reported by UOS) represent an anomalous impacted zone on the property, identifies an authentic region of high arsenic or lead concentration (hot spot) or accurately represents the average metals concentration observed for the entire property.

3) Evaluate the spatial distribution of arsenic and lead concentrations in the surface soil of contiguous yards in order to judge whether lead and arsenic concentrations are similar or dissimilar from the target residence.

Study Objective #1-2: Choose 3 residences within the study area, identified by the UOS investigation, which are below the removal action levels (defined as unimpacted) (USEPA 1998b). Collect surface soil samples on a 5'x 5' grid to characterize the spatial relationship of metals (As, Cd, Pb, Zn) concentrations at the property. Whenever possible, this grid sampling will extend out approximately 15 feet to properties adjacent to the targeted residences where yards are contiguous. This information will be used to:

1) Determine the mean metals concentrations for the front and back yards at each residence. Evaluate whether concentrations differ significantly between front and back yards.

2) Evaluate the spatial distribution of arsenic and lead concentrations at the unimpacted residence in order to judge whether the grab sample concentrations for each residence (reported by UOS) represent an anomalous unimpacted zone on the property, identifies an authentic region of low arsenic or lead concentration or accurately represents the average metals concentration observed for the entire property.

3) Evaluate the spatial distribution of arsenic and lead concentrations in the contiguous yards in order to judge whether lead and arsenic concentrations are significantly similar or dissimilar from the unimpacted residence.

Study Objective #1-3: Collect four core samples (2 front yard cores and 2 backyard cores) from each of the 5 target residences and 3 unimpacted residences. Core samples will be 2-12 inches in depth and will be used to determine depth profile and to evaluate if buried sources may exist. The core sample will be fractioned into 2-inch intervals and each depth interval will be containerized separately. These samples will be analyzed for arsenic, cadmium, lead and zinc and then archived for possible phase speciation and particle sizing. The decision to perform additional analyses will be made by the RPM and Regional Toxicologist after preliminary core soil results are available. This information will be used to:

1) Determine what arsenic, cadmium, lead and zinc concentrations are present in each core sample.

2) Determine whether visual inspection of the core uncovers an observed stratification of soil contamination, for example: native soils stratified with other types of fill material.
3) Using phase speciation and particle sizing, determine potential sources of soils present at the residence.

Study Objective #1-4: After the initial analysis of all surface and core soils (discussed in Study Objectives #1-1 to #1-3) which quantified arsenic, cadmium, lead and zinc concentrations in each sample, a subset of approximately 20% ($N \geq 30$) of surface soils and approximately 30% ($N \geq 7$) of core samples will be identified for analysis for a full suite of PCOC metals (Section B4.2). This information will be used to:

1) Determine if any metals are present in quantities above proposed health-based goals (Section B4.2.1).
2) Determine what, if any, metals are useful indicators for source attribution.

Overall Study Goal #1 Data Evaluation:
Data from each of the impacted and unimpacted residences will then be compared to determine the following:

1) Determine whether the mean arsenic, cadmium, lead and zinc concentrations found at impacted and unimpacted residences are statistically different.
2) Determine if any single residence or group of residences report mean arsenic and/or lead concentrations for either the front yard or the back yard above the removal action levels.

STUDY GOAL #2
Study Goal #2 will be carried out only after residences have been identified for soil removal. This will be determined by an investigation performed by the On-Scene Coordinator that is not part of the scope of this Project Plan.

Study Objective #2-1: Collect indoor household dust and undisturbed dust samples from each residence identified for soil removal. Household dust samples will be collected in the main living space of the residence. Undisturbed dust samples will be collected in the attic (if it is not used as a living space). All samples will be analyzed for arsenic, cadmium, lead and zinc by XRF. After analysis, all samples will be archived for possible future lead and arsenic speciation and particle sizing analyses or quantification of additional metals. The decision to perform additional analyses will be made by the RPM and Regional Toxicologist after preliminary concentration data are available. Standard operating procedures for speciation and particle sizing are located in Appendix A.

Study Objective #2-2: Using the same residences chosen for Study Objective #2-1, collect first morning flush and post-flush tap water samples from each residence which will be analyzed for lead.

Study Objective #2-3: Using the same residences chosen for Study Objective #2-1, perform field screening to quantify the levels of lead paint present on indoor and outdoor surfaces of target homes.

Overall Study Goal #2 Data Evaluation:
These environmental data will be used to quantify potential sources of arsenic or lead exposure and to improve estimates of risk to humans. Data will also be used to characterize levels of cadmium and zinc present in these media. In addition, these data will be used to refine the fate and transport component of the conceptual site model by investigating the relationship between:

- soil-arsenic vs. house dust-arsenic
- soil-arsenic vs. attic dust-arsenic
- soil-lead vs. house dust-lead
- soil-lead vs. attic dust-lead
- paint-lead vs. house dust-lead
- paint-lead vs. attic dust-lead
- soil-metal vs. house dust-metal (metal = cadmium or zinc)
- soil-metal vs. attic dust-metal (metal = cadmium or zinc)

STUDY GOAL #3

Study Objective #3-1: Using the same residences chosen for Study Goal #2 as the population group, measure the levels of arsenic present in composite hair samples and first morning void urine samples for all willing members of each household. These data will be used to estimate whether acute or subchronic exposures to arsenic have potentially occurred, and if so, to what extent.

Study Objective #3-2: Using the same residences chosen for Study Goal #2 as the population group, collect blood lead samples from children ages 6-72 months (with parental consent). These data will be used to estimate the actual lead exposure levels in the children. Because the number of children present at these residences is presently unknown, it is uncertain whether a sufficient number of children will be recruited to perform a meaningful quantitative evaluation of the mean blood lead values for this study region. In the event that a meaningful statistical evaluation of blood lead values for the Vasquez Boulevard and I-70 site community cannot be determined, these data will be used only to judge if a particular child's blood lead concentration is above a level of health concern.

Study Objective #3-3: Using the same residences chosen for Study Goal #2 as the population group, administer in-home questionnaires that will be used to provide additional information pertinent to evaluate potential arsenic and lead exposures.

Overall Study Goal #3 Data Evaluation:

This information will be used to determine whether arsenic or lead exposures to residents within the study area are significantly:

a) higher than national averages or exceeds EPA guidelines for child blood lead values; or
b) above a level of health concern

As a secondary goal, the biological data will be used in tandem with demographic and environmental data to quantify the following relationships, providing sufficient data are available:

1) Determine whether a statistically significant relationship ($\alpha > 0.95$) is present between:

   - soil-arsenic vs. urine-arsenic
   - soil-arsenic vs. hair-arsenic
   - soil-lead vs. blood-lead
   - house dust-lead vs. blood-lead
   - attic dust-lead vs. blood-lead
   - paint-lead vs. house dust-lead
   - paint-lead vs. attic dust-lead

2) This information may be used to determine whether a quantitative or semi-quantitative relationship exists for the following. Other comparisons may be made as necessary.

   - blood-lead vs. mouthing habits

A7 DATA QUALITY OBJECTIVES

The DQO process is an iterative process which is designed to focus on the decisions that must be made and to help ensure that the site activities acquire data that are logical, scientifically defensible, and cost effective. The DQO process is intended to:

- Ensure that task objectives are clearly defined
- Determine anticipated uses of the data
- Determine what environmental data are necessary to meet these objectives
- Ensure that the data collected are of adequate quantity and quality for the intended use

Two types of DQOs are identified in this SAP/QAPP: DQOs for the overall study

objectives and criteria for measurement data. DQOs for the overall study objectives address the first three steps in the DQO process described above. These DQOs have been addressed in Section A6.2. DQO requirements that ensure data of sufficient quantity and quality are obtained are presented in the following section.

Criteria for Measurement Data

The performance criteria for measurement data generated as part of this project will be evaluated in terms of precision, accuracy, representativeness, completeness and comparability (PARCC). The following sections describe PARCC criteria. DQO criteria required for each study objective is also provided.

Precision: Precision is defined as the agreement between a set of replicate measurements without assumption or knowledge of the true value. It is a measure of agreement among individual measurements of the same property under prescribed similar conditions. Agreement is expressed as the relative percent difference (RPD) for duplicate measurements if the reported values are sufficiently above the method detection limit (MDL) (> 5 x MDL) or the absolute difference of two values near the MDL.

$$RPD = \frac{|\,2\,(A - B)\,|}{A + B} \times 100\%$$

$$\text{Absolute difference} = |\,A - B\,|$$

Where:

A = original concentration value of an analyte
B = duplicate concentration value of an analyte

Additionally, agreement may be expressed as the range and standard deviation for larger numbers of replicates. The appropriate precision calculation will be reported for the required duplicates, and a defined MDL will be reported as per EPA guidance in 40 CFR, part 136, Appendix B.

Field Duplicates: Field duplicates are co-located samples that are collected at the site. These samples are submitted blind to the laboratory to test both the precision of the laboratory analysis in conjunction with the precision of sample collection. Field duplicates are required to be collected at a minimum frequency of 5% of all surface soil samples collected (1 field duplicate per 20 investigation samples collected). The RPD for field duplicates should not exceed requirements outlined in Table A7.1 or, alternatively, the absolute difference should not exceed 1 x MDL. However, these acceptance limits may be arbitrary; therefore, a graphical comparison of the original and field duplicate samples should also be prepared. This comparison will include a linear regression and will report the calculated correlation coefficient (r).

Laboratory Duplicates: Laboratory duplicates are splits that are prepared in the laboratory. Because the laboratory is aware that the samples are duplicates, these samples serve to test the precision of the laboratory's sample preparation and analysis. The RPD for laboratory duplicates should not exceed requirements outlined in Table A7.1 or, alternatively, the absolute difference should not exceed 1 x MDL.

Confirmation Samples: Confirmation samples will be analyzed by XRF and confirmed using another metals analysis performed by an independent laboratory. Confirmation analyses will be performed on 20% of surface soils collected. The RPD for confirmation samples should not exceed requirements outlined in Table A7.1 or, alternatively, the absolute difference should not exceed 1 x MDL. However, these acceptance limits are arbitrary; therefore, a graphical comparison of the XRF analysis and the corresponding metals analysis should also be prepared. This comparison will include a linear regression and will report the calculated correlation coefficient (r). Due to limited samples available for soil cores, household dust, attic dust, tap water, hair and urine samples, these media will not require confirmation analyses by an independent laboratory.

Table A7.1: Precision requirements for each Study Objective

Study Objective	Project QC or Laboratory QC	Precision Test	Frequency	Test Criteria
#1-1	Project QC	Field duplicates	5% of investigative samples collected (1 field duplicate per 20 investigative samples collected)	RPD < 25%[a]
	Project QC	XRF sample vs. Independent Metals Analysis Sample	20% of investigative samples (1 confirmation sample/5 investigative samples)	Prepare graphical presentation of XRF vs. Metals samples which reports a linear regression
	Laboratory QC	XRF Method duplicates	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
	Laboratory QC	Independent Metals Analysis duplicates	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
#1-2	Project QC	Field duplicates	5% of investigative samples collected (1 field duplicate per 20 investigative samples collected)	RPD < 25%[a]
	Project QC	XRF sample vs. Independent Metals Analysis Sample	20% of investigative samples (1 confirmation sample/5 investigative samples)	Prepare graphical presentation of XRF vs. Metals samples which reports a linear regression

Study Objective	Project QC or Laboratory QC	Precision Test	Frequency	Test Criteria
	Laboratory QC	XRF Method duplicates	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
	Laboratory QC	Independent Metals Analysis duplicates	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
#1-3	Laboratory QC	XRF Method duplicates	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
	Laboratory QC	Independent Metals Analysis duplicates	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
#1-4	Laboratory QC	Independent Metals Analysis duplicates	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
#2-1	Laboratory QC	Method duplicates for indoor household dust	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
	Laboratory QC	Method duplicates for attic dust	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]

Study Objective	Project QC or Laboratory QC	Precision Test	Frequency	Test Criteria
#2-2	Laboratory QC	Method duplicates for tap water	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 20%[b]
#2-3	Project QC	Field duplicates for lead paint	5% of homes screened (1 duplicate per 20 investigative samples)	RPD < 25%[a]
#3-1	Laboratory QC	Method duplicates for urine arsenic	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
	Laboratory QC	Method duplicates for hair arsenic	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 25%[b]
#3-2	Laboratory QC	Method duplicates for blood lead	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	RPD < 20%[b]
#3-3	Project QC	Have team member review the interview sheets to ensure all information has been obtained	100% of the completed interview sheets	N/A

N/A – Not applicable

a – Perform absolute difference calculation as described above for analytical results near the MQL. Also prepare a graphical presentation which reports the linear regression.

b – Perform absolute difference calculation as described above for analytical results near the MQL.

Accuracy: Accuracy is a measure of the closeness of individual measurements to the "true" value. Accuracy usually is expressed as a percentage of that value. For a variety of analytical procedures, standard reference materials traceable to or available from National Institute of Standards and Technology (NIST) or other sources can be used to determine accuracy of measurements. Accuracy will be measured as the percent recovery (%R) of an analyte in a series of reference standards that span the linear range of the instrument. Specific accuracy guidelines for other accuracy measurements such as calibration verification standards are detailed in the SOPs (See Appendix A).

$$\%R = \frac{A}{B} \times 100\%$$

Where:

A = measured concentration value of an analyte
B = true (known) concentration value of an analyte

Accuracy will be measured by performing analysis of matrix spike (MS) samples and laboratory control samples (LCSs).

Matrix Spike: A matrix spike sample is an investigative sample having a matrix that is representative of all investigative samples to which a known concentration of target analytes is added. This quality control sample measures the extent that the sample matrix affects the accuracy of reported target analytes.

Laboratory Control Sample: A LCS originates in the laboratory or is provided as a standard reference material (SRM) by a manufacturer (eg. NIST) and contains target analytes of known concentration. Because LCSs are independent of the calibration standards, they are analyzed to verify the accuracy of the standards used to calibrate the instrument. At least one LCS must be analyzed in each analysis batch and analytical results must fall within manufacturer's limits.

Table A7.2: Accuracy requirements for each Study Objective

Study Objective	Project QC or Laboratory QC	Accuracy Test	Frequency	Test Criteria
#1-1	Laboratory QC	MS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	75-125 %R
	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits
#1-2	Laboratory QC	MS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	75-125 %R
	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits
#1-3	Laboratory QC	MS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	75-125 %R
	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits
#1-4	Laboratory QC	MS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	75-125 %R
	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits

Study Objective	Project QC or Laboratory QC	Accuracy Test	Frequency	Test Criteria
#2-1	Laboratory QC	MS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	75-125 %R
	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits
#2-2	Laboratory QC	MS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	75-125 %R
	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits
#2-3	Project QC	LCS	5% of homes screened (1 duplicate per 20 investigative samples)	80-120 %R
#3-1	Laboratory QC	MS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	75-125 %R
	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits
#3-2	Laboratory QC	LCS	5% of investigative samples per analytical batch (1 duplicate per 20 investigative samples)	Manufacturer's Limits
#3-3	N/A	N/A	N/A	N/A

MS – Matrix Spike
LCS – Laboratory Control Sample
%R – Percent Recovery
N/A – Not applicable

Representativeness: Representativeness is defined as the degree to which data accurately and precisely describe the general characteristics of a population or the parameter variations at a sampling point. It is important to determine whether samples collected for this investigation are representative at both levels.

Representativeness for the Sample Population: The sample populations chosen to fulfill each Study Goal have been selected using various sampling strategies. Details and rationale for each sample population are discussed below.

Study Goal #1:

Impacted Residences

A biased sampling strategy has been chosen to investigate 5 residences where soil removal actions may be necessary. This sampling strategy will serve to produce a sample population that will be representative of those residences where highest lead and arsenic concentrations were reported by UOS. This quantity of residences (N=5) was chosen in order to generate sufficient data to perform meaningful statistical evaluations of results while minimizing the analytical costs related to this intensive sampling effort.

Unimpacted Residences

A pseudo-random sampling strategy, stratified into 3 concentration ranges below the removal action level for arsenic (As < 400 ppm), has been chosen to be investigated for 3 unimpacted residences. One sample will be randomly selected from each of the following concentration ranges:

Arsenic Concentration Range (ppm)
$44 \leq [As] < 100$
$100 \leq [As] < 200$
$200 \leq [As] < 400$

Samples will be randomly chosen within each concentration range must not fall on the same streets for which Impacted Residences have been chosen. These residences have been chosen to produce a sample population (N=3) that will be representative of those properties where soil removal actions are not anticipated to be required.

Soil Cores

Core samples will be randomly identified within each section (front yard or back yard) of the residence planned for sampling. Because the residences sampled will be those where both the highest and lowest arsenic and lead levels are expected, this biased sampling strategy should produce a sample population (N=32) that will be representative of the site.

PCOC Metals Quantification

When arsenic results for surface and core soils obtained during this investigation are available, these soil samples will be stratified into arsenic concentration

ranges. Samples identified for further metals characterization will be randomly selected from each of four concentration ranges such as the example presented below.

Category	Concentration Range
Low	$0 < [As] < 200$ ppm
Medium-Low	$200 \leq [As] < 600$ ppm
Medium-High	$600 \leq [As] < 1000$ ppm
High	$[As] \geq 1000$ ppm

This pseudo-random, stratified sampling strategy will serve to produce a sample population that will be representative of the site.

Study Goal #2:
A biased sampling strategy has been chosen to investigate potential sources of arsenic, cadmium, lead and zinc in various environmental media where removal actions are planned. This sampling strategy will serve to produce a sample population that will be representative of those residences where elevated metals concentrations are likely to be present.

Study Goal #3:
A biased sampling strategy has been chosen to investigate biological media for the purpose of determining the extent to which acute or subchronic lead or arsenic exposure may be observed in homes where soil removal is planned.

Comparability: Data are comparable if collection techniques and measurement procedures are equivalent for the samples within a sample set. Comparable data will be obtained by specifying standard units for physical measurements and standard procedures for sample collection, processing, and analysis. See the attached SOPs (Appendix A) for sampling and for analytical procedures.

Completeness: Data are considered complete when a prescribed percentage of the total measurements and samples that are planned are actually obtained.

Data Collection (except analytical data): Due to the limited quantity of residences planned for environmental sample collection, every effort will be made to collect all the data prescribed within this SAP/QAPP; however, a minimum completeness goal will not be identified. Likewise, efforts will be made to maximize participation in the biomonitoring and demographic data collection while minimizing the nuisance to the residents. It is therefore, not reasonable to prescribe a minimum completeness goal for these aspects of the project. However, any data gaps encountered and the potential impact of the gaps will be discussed in the report detailing findings (Section D2.2).

Analytical Data Produced by Laboratories: Data, produced by an analytical laboratory, must be valid for at least 90% of analyzed samples. This means that

fewer than 10% of all analytical data generated for each analysis method may incur a qualification of unusable (R qualification). If this completeness goal is not met, the analytical laboratory responsible for generating the poor quality data must reanalyze samples without additional cost and reanalyses must adhere to method requirements to generate valid data.

Detection Limits (applicable to chemical analyses only): MDLs are minimum concentration of a substance that can be measured and reported with 99% confidence that the true value is greater than zero (3σIDL). The method quantitation limits are the minimum values that can quantify that analyte with reasonable scientific confidence (10σ IDL). The method quantitation limits established for the analytical methodologies to be employed for this effort are presented in the next section (Section B).

A9 SPECIAL TRAINING REQUIREMENTS AND CERTIFICATION

Personnel responsible for completing this project include toxicologists, analytical chemists, phlebotomists and geologists. These technically-trained personnel have been chosen to participate in the investigation because they are experienced in conducting sampling programs, chemical measurements on a variety of analytical instrumentation and performing interpretation of data generated from the sampling program.

Personnel retained for field sampling activities must be OSHA 40 Hour HAZWOPER certified. Field sampling personnel must also be familiar with the sampling protocols (SAP/QAPP) and must ensure that all project requirements for sampling are met. Personnel retained for blood collection must be a certified pediatric phlebotomist.

A10 DOCUMENTATION AND RECORDS

Maintenance of pertinent documentation is critical for evaluating the success of the investigation. This section describes the laboratory requirements for preparing data packages for this project. In addition, procedures for storing and maintaining laboratory data are described in this section. Documentation describing sample handling and custody requirements are discussed in Section B3 of this SAP/QAPP.

A10.1 Laboratory Data

Contract Laboratory Program (CLP)-like data packages will be required for all laboratory analytical data. These CLP-like data packages will include a case narrative, copies of all associated raw data, sample results and all associated QC summaries. A summary of the data package requirements is shown on the next page:

Section I **Case Narrative**

1. Case narrative
2. Copies of nonconformance/corrective action forms
3. Copies of sample receipt notices
4. Internal tracking documents, as applicable
5. Copies of all chain-of-custody forms

Section II **Analytical Results** - All results will be reported on a dry weight basis.

1. Results for each parameter including dilutions and reanalysis (dry-weight basis)
2. Units of measure
3. Method Quantitation Limit
4. Date of sample analysis
5. Date of sample receipt
6. Date of sampling
7. Dilution factor

Section III **QA/QC Summaries**

1. Method blanks, continuing calibration blanks, preparation blanks
2. Initial and continuing calibration verifications
3. Inductively Coupled Plasma (ICP) interference check samples
4. Matrix spikes and post-digestion spikes
5. Method duplicate samples
6. Laboratory control samples
7. Method of standard additions
8. ICP serial dilution
9. Instrument detection limits

Section IV **Instrument Raw Data** – Sequential measurement readout records for XRF, ICP, graphite furnace atomic absorption (GFAA), which will include the following information:

1. Environmental samples, including dilutions and reanalyses
2. Initial calibration (including reporting whether $r \geq 0.995$)
3. Initial and continuing calibration verifications
4. Method blanks, continuing calibration blanks and preparation blanks
5. ICP interference check samples
6. Matrix spike and post-digestion spikes
7. Matrix duplicate samples
8. Laboratory control samples
9. Method of standard additions
10. ICP serial dilution

Section V **Other Raw Data**

1. Sample digestion and preparation logs
2. Instrument analysis logs for each instrument used
3. Standard preparation logs, including initial and final concentrations for each standard used

Section VI **Electronic Data** – All analytical data will be supplied in electronic form as well as hardcopy form. All data will be provided in an Office '97 Excel® spreadsheet. An example spreadsheet format has been developed and is attached (Figure A10.1).

A10.2 Data Management

Hardcopy Data

Hardcopies of analytical data will be provided by all analytical laboratories. These data will be reviewed by ISSI and a copy provided to EPA for their records.

Electronic Data

Electronic data will be provided by all ISSI subcontractors in Office '97 Excel® spreadsheet formats provided in Figure A10.1. These data will be verified with hardcopy analytical results to ensure no transcription errors have occurred. These data will then be imported into and maintained in an Access® database or an Excel® spreadsheet. The database or spreadsheets will be used by ISSI to perform statistical calculations and trend evaluations. Results of database queries will be incorporated into a report, described in Section D2.2, which will be submitted to EPA's Regional Toxicologist, Dr. Chris Weis.

B MEASUREMENT AND DATA ACQUISITION

This section describes the site investigation design and implementation, including method for sample collection, handling and analysis. In addition, field and laboratory QC procedures and instrument testing, inspection, maintenance and calibration requirements are described.

B1 SAMPLING PROCESS DESIGN

The sampling process design is described in this section. Sampling process design includes descriptions of the sampling locations, number of samples planned for collection, sample matrices and the measurement of field parameters.

B1.1 Identification of Sample Locations

Rationale for sample location identification may be divided into two portions: 1) grid sampling for Intensive Surface and Core Soil Sampling Phase; and 2) sample locations for Environmental and Biomonitoring Sampling Phase. Each of these is described below.

FIGURE A10.1: XRF/Wet Chemistry Results

STATION NO.	TAG NO.	Lab Sample ID	Date Extracted	Date Analyzed	Analyte Name	Dilution	Qualifier	Result	Method Detection Limit	Project Required Detection Limit	Units

Grid Sample Locations

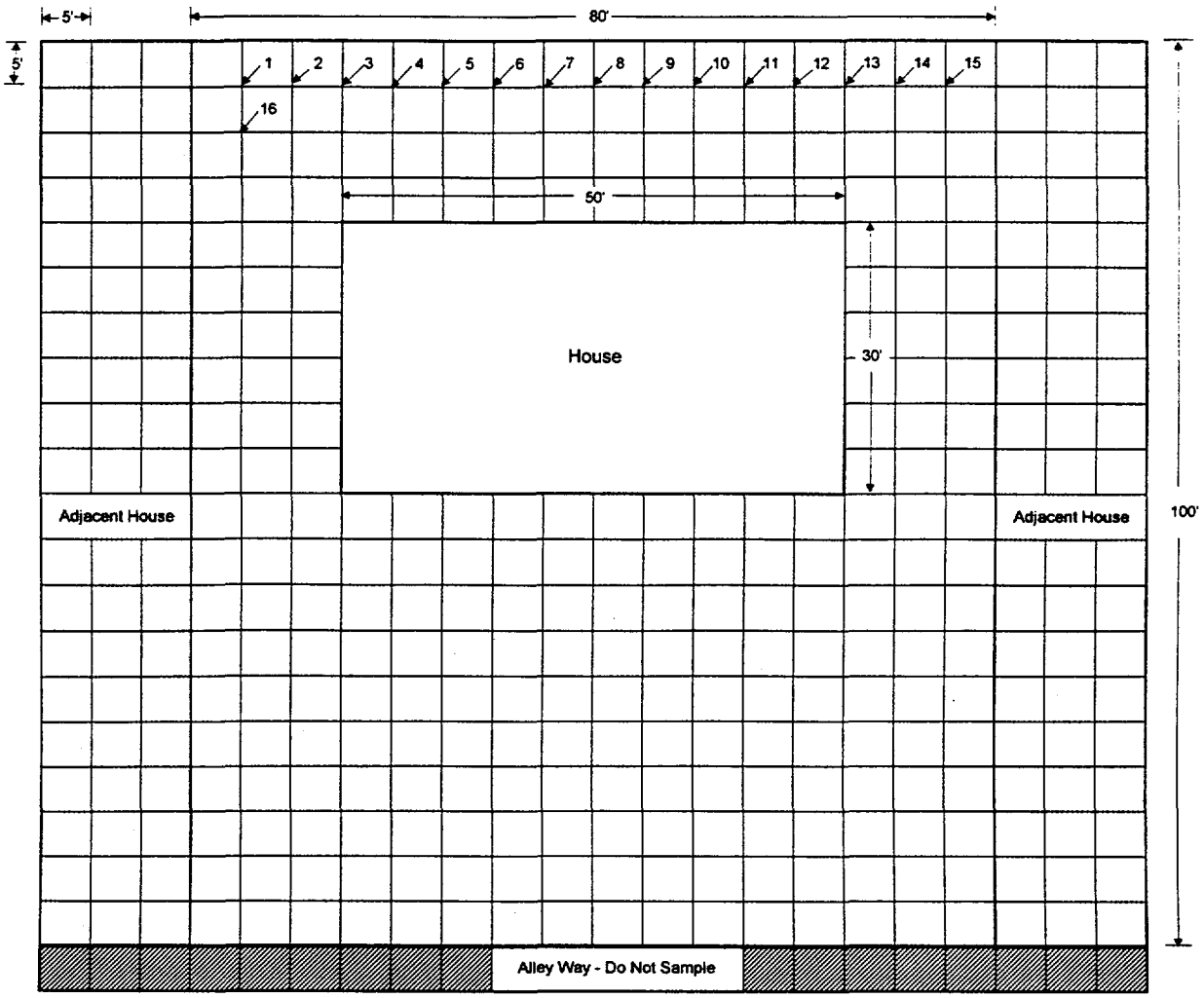
Eight residences (5 impacted and 3 unimpacted) have been identified for intensive grid sampling (Table B1.1.1). Surface soil samples will be collected in the adjacent yards providing residents will grant permission for the sampling teams to gain access. A 5'x 5' grid will be imposed over the residence and 15 feet into adjacent residences having contiguous yards. Samples will not be collected from the alleyways at the rear of the residences. Surface soil samples will be collected at each grid node or the point closest to that grid node if obstructions are noted. Figure B1.1.1 presents the proposed sampling design for intensive surface soil sampling. In addition, 4 soil cores will be collected at the residence, 2 cores in the front yard and 2 cores in the back yard.

Individual samples will be identified using the following procedure:

- Field personnel will draw a picture of the target residence and the adjoining properties in the field logbook. The 5' x 5' grid will be superimposed over the drawing of the residence.
- Surface soil samples will be individually numbered and these numbers will be noted on the drawing in the field logbook.
- Each sample number will be identified using a four-part sampling code as described in Section B2.2.

Numbering assignments will begin at first grid node located at the northwesternmost corner of the yard. This sample will be identified as 001. Sample numbers will increase going east (002, 003, 004, etc.) until there are no more grid nodes on that row identified for sampling at the residence. The sample numbering will continue on the next row of grid nodes directly south of the first grid node (001) and will increase again going east until there are no more grid nodes on that row. Numbering will continue until there are no more grid nodes identified for sampling on the residence. Sample number assignments for adjoining residences will be identified as a unique samples (since the address is different) and will be assigned using the sample protocols described above.

FIGURE B1.1.1 5' X 5' GRID SAMPLING DESIGN



Residence dimensions are estimates.

In order to maintain confidentiality, the addresses for residences slated for this investigation have been assigned a Residence Code. The 8 residences identified for intensive soil sampling are provided in Table B1.1.1.

Table B1.1.1: Residences Identified for Intensive Surface Soil and Core Sampling

Target Residence Definition	Residence Code	Arsenic Concentration[a] (ppm)
Impacted	A	5600
	B	530
	C	1700
	D	2600
	E	780
Unimpacted	F	98 J
	G	140 J
	H	350

a – Highest concentration measured at each property.

Environmental and Biomonitoring Sample Locations

This phase of sampling will proceed after residences requiring soil removal action are identified. The arsenic or lead surface soil concentrations above removal action levels for the 37 residences will be confirmed. Any residences where arsenic and lead concentrations are confirmed to be greater than 400 ppm arsenic and/or 2000 ppm lead will be scheduled for removal actions. All residences, identified for soil removal action, will be sampled or field analysis performed for the following environmental and biologic media before any soil is removed from the residences.

Environmental Samples:

- field analysis of exterior and interior paint (lead only)
- indoor household dust (arsenic, cadmium, lead)
- attic dust (arsenic, cadmium, lead)
- tap water (lead only)
- garden vegetables (arsenic, cadmium, lead)
- surface soil samples co-located with garden vegetables (arsenic, cadmium, lead)

Biological Samples:

- Arsenic in composite hair samples
- Inorganic arsenic in first void urine samples
- Blood lead

B1.2 Measurement of Field Parameters

Analysis of exterior and interior paint screening will be performed using portable XRF lead paint analyzers on painted surfaces including interior and exterior walls and trim. The lead paint analyzer must be calibrated everyday prior to use at each residence. All calibration and analytical measurements must be documented in the field logbook. Calibration procedures and lead paint measurements will be performed in accord with the SOP (Appendix A).

B2 SAMPLING METHOD REQUIREMENTS

The following sampling method requirements will be discussed in this section:

- Identification of sampling protocols to be used
- Field sample identification procedures
- Decontamination procedures

B2.1 Sampling Protocols

Samples will be collected according to SOPs provided in Appendix A. Procedures outlined in these SOPs include:

- Surface Soil Sampling and Sieving
- Tap Water Collection
- Indoor Household Dust Collection
- Indoor Undisturbed Dust Collection
- Interior/Exterior Paint Screening
- Blood Lead Sampling
- Collection of Urine Samples for Arsenic
- Collection of Hair Samples for Arsenic
- Collection of Garden Vegetables

Soil Core Samples

Because surface soil to a depth of 12 inches is presently too dry to support standard coring methods, collection of soil cores will be performed by digging a pit to a depth of 12 inches. Soil core depth of 12 inches has been chosen because this is the maximum depth to which any soil removal actions will occur. After the pit is opened, a photograph will be taken. The photograph will serve to document any soil stratification observed for that core. The photograph will be given an identification number and both a description of the photograph and the identification number will be noted in the field logbook. The number for each photo will be the same sample identification number assigned to the soil core. If a distinct stratification of soil material is noted during soil core collection (eg. native material vs. fill material), the soil will be divided into 2 portions along the observed soil stratification.

Surface Soil Samples Co-located with Garden Vegetables

Surface soil samples that are co-located with any garden vegetables that are collected will also be obtained. The SOP for Surface Soil Sampling and Sieving (Appendix A) must be followed using the guidelines for surface soil compositing. A composite sample of garden soil consisting of 6 subsamples will be collected. The 6 subsamples will be collected along the entire vegetable bed.

A summary of required sample containers, preservation, analytical instrumentation and holding times is presented in Table B2.1.1.

Table B2.1.1: Summary of Sample Containers, Preservation, Analysis Methods and Holding Times

Sample Media Collected	Target Analytes	Sample Container	Sample Preservation	Sample Holding Time[a]	Analytical Instrumentation
Environmental					
Indoor House Dust	As, Cd, Pb	Filter Cartridge	None.	180 days	XRF
Undisturbed Dust	As, Cd, Pb	Filter Cartridge	None.	180 days	XRF
Tap Water	Pb	1 500-mL HDPE bottle 1 1-L HDPE bottle	~1 mL Conc. HNO_3 acid (pH<2)	180 days	GFAA
Interior & Exterior Paint	Pb	N/A	N/A	N/A	Portable XRF
Garden Vegetables	As, Cd, Pb	Zip-lock Storage bags	None.	180 days	GFAA
Surface Soil Samples Co-located with Garden Vegetables	As, Cd, Pb	4-oz wide mouth glass jar or zip-lock bags	None.	180 days	XRF
Biological					
Hair	As	Plastic vial w/ screw-on cap	None.	180 days	Hydride Generation AA
Urine	As	Plastic, sterile urine cup	Refrigerate		Hydride Generation AA
Blood	Pb	6-mL lavender top Vacutainer™ tube	EDTA, Refrigerate		GFAA

XRF – X-ray Fluorescence
GFAA – Graphite Furnace Atomic Absorption
HDPE – High Density Polyethylene
HNO_3 – Nitric
N/A – Not applicable
AA – Atomic Absorption
a – Holding time is calculated from sampling date.

B3 SAMPLE HANDLING AND CUSTODY REQUIREMENTS

B3.1 Chain-of-Custody

Rationale: To ensure that collected samples are properly tracked and maintained in proper custody.

All samples collected in the field will be submitted to the appropriate analytical laboratory under chain-of-custody. All sample transfers must adhere to chain-of-custody procedures detailed below.

Sample custody history of each sample and its handling will be documented on a chain-of-custody (COC) form covering all transfers of custody until arrival at the analytical laboratory. These forms are prepared in triplicate on carbonless forms. Each COC form will identify the samples included in the sample delivery group (SDG) and the required analyses. The following information should be included on each COC form:

Client Name -	Enter ISSI, Inc.
Address -	Enter 999 18th Street, Suite 1180; Denver, CO 80202
Phone No. -	Include the phone number of the contact at ISSI (292-4142 x235).
Fax No. -	Include the facsimile number of the contact at ISSI (292-4926).
Date -	Enter the date the COC form was prepared.
Page -	Indicate page number and total number of COC pages in the SDG.
Project Name -	Enter the name of the project (Vasquez Boulevard and I-70 Residential Risk-based Sampling – Stage I Investigation).
Send Report to -	Indicate the contact name to whom the report should be sent (Mary Goldade).
Sampler Name/Signature -	Sampler's name.
Sample Number -	Note each discrete sample ID included in the SDG.
Sampling Date/Time-	Enter the specific date and time the sample was taken.
Preservative -	Indicate the type of preservative used. If no preservative was required, write "none".
Container Size/Type -	Indicate the container type and size (where applicable).
Sample Description-	Mark the sample matrix.
Analysis Required -	Indicate the method reference and name of analyses required. In the boxes below, mark an "X" to indicate the analysis is required for the respective sample ID.
Comments -	Any notes of interest (sample condition, etc.) are entered here.
Relinquished by-	The person transferring the samples signs his name here.
Date-	The person transferring the samples enters the date of relinquishment.
Company-	The person transferring the samples enters his company name.
Time-	The person transferring the samples enters the time relinquished.
Received by-	The person accepting the samples signs his name here.
Date-	The person accepting the samples enters the date of relinquishment.
Company-	The person accepting the samples enters his company name.
Time-	The person accepting the samples enters the time relinquished.

Each complete COC form will be reviewed for accuracy and clarity by the sampler and then signed by the sampler or On Scene Coordinator prior to sample shipment or transfer of custody. When the samples are handed over to a designated lab courier, the courier will compare sample inventory with the COC form to ensure accuracy. The COC forms are then signed by the courier to serve as written acknowledgment that the samples have been transferred in tact to the courier. The sampler will be given a copy of the COC form with release signatures. One copy will be retained by COC form initiator. When the samples arrive at the laboratory, the lab's sample custodian will document the date and time of receipts and condition of the samples (temperature of samples, note any damage, etc.).

Third party custody will be required when samples are shipped. Third parties include shipping companies such as FED EX, UPS and USPS. Samples will be shipped overnight in tightly sealed ice chests. All packing procedures will conform to appropriate IATA and/or DOT requirements. Third party couriers or clerks will not sign for relinquishment on COC forms. Instead, copies the shipping/tracking forms will be retained as documentation of transfer of custody. The COC form which corresponds to the samples being shipped will be sealed inside the shipping container but inside a plastic zip-lock bag and taped to the cooler lid to avoid water damage from ice.

All corrections to the chain-of-custody record will be initialed and dated by the person making the corrections. Each chain-of-custody form will include signatures of the appropriate individuals indicated on the form. The originals will accompany the samples to the laboratory, and copies documenting each custody change will be recorded and kept on file.

Chain-of-custody will be maintained until final disposition of the samples by the laboratory and acceptance of analytical results by EPA.

B3.2 Field Documentation

All sampling procedures will be documented in a field logbook. These general guidelines for maintaining field documentation will be used:

- Documentation will be completed in permanent black or blue ink.
- All entries will be legible
- Errors will be corrected by crossing out with a single line, dating and initialing the lineout.

Field personnel will use bound field logbooks with sequentially numbered pages to maintain field records. The following information will be recorded in the field logbook:

- Name and affiliation of all personnel or visitors on site
- Weather conditions during the field activity
- Chronology and summary of daily activities
- Notes of conversations with coordinating officials
- Identification numbers of field instruments used
- Results of calibrations and field measurements
- Documentation of sampling activities, including data and time of sample collection and names of sampling personnel
- Decontamination episodes
- Reference to other field logbooks or forms that contain related information
- Discussion of problems encountered and the resolution obtained
- Discussion of deviation from the SAP/QAPP
- Description of any photographs taken, including date, time, direction, photo ID and photographer
- Record of QC samples collected

B3.3 Sample Archives

All surface soil, soil cores and dust samples must be retained in a dry and secure storage facility. A portion of samples may be identified for further characterization; therefore samples must be stored in such a manner that quick retrieval is possible. All investigative samples will be held in storage, under chain-of-custody until the RPM indicates that these samples may be disposed according to proper waste disposal methods.

B4 ANALYTICAL METHOD REQUIREMENTS

This section provides the details necessary to prepare and analyze surface soil samples to meet project objectives and quality control (QC) requirements. Methods described in this section include: sample preparation and metals analysis for a variety of media.

B4.1 Sample Preparation

All surface soils must be prepared before any analytical measurements are made. Soils will be air-dried for a minimum of 8 hours prior to sieving. Following drying each sample must be sieved into a fine fraction. Fines are sieved to a grain size of less than 250 µm. Procedures for sieving and decontamination of sieving equipment are outlined in the standard operating procedures (SOP) located in Appendix A.

B4.2 Metals Analysis

The primary method for quantification of arsenic and lead for surface soils will be via XRF. Twenty percent of samples identified for XRF analysis will also be confirmed using another analytical method. Specifically, samples identified for confirmation

analyses will be sieved into a <250 μm size fraction, then split and submitted to a contract laboratory for metals analysis. Analysis of tap water, dust, blood lead, urine arsenic, hair arsenic, garden vegetables and surface soils co-located with garden vegetables will be submitted to an analytical laboratory for standard metals analyses. Due to anticipated limited sample volumes, these samples will not require confirmation analyses.

B4.2.1 Detection Limit Requirements

A preliminary health-related goal (HRG) concern for a resident at a cancer risk of 1E-4 was calculated for arsenic. The preliminary HRGs for all metals but arsenic was determined based upon a hazard quotient (HQ) equal to 1. These values are presented in Tables B4.2.1 and B4.2.2. A detailed summary of the calculations for the health-related goals is presented in Appendix B. All analytical methods utilized must be able to achieve the method quantitiation limits (MQLs) for all target analytes provided below.

Table B4.2.1 Health-Related Goals and Target Method Quantitation Limits for Environmental Samples

Target Analyte	Risk-based Assumptions	Health-related Goals		Method Quantitation Limit (MQL)	
		Soil/Solid[a] (mg/kg)	Water (mg/L)	Soil/Solid[a] (mg/kg)	Water (mg/L)
Environmental Samples – Standard 5 Metals					
Arsenic	Risk = 1E-4	35	N/A	10	N/A
Cadmium	HQ = 1	92	N/A	10	N/A
Lead	HQ = 1	400	10	40	0.002
Zinc	HQ = 1	28,000	N/A	25	N/A
Environmental Samples – PCOCs Evaluation					
Aluminum	HQ = 1	92,000	N/A	5	N/A
Antimony	HQ = 1	37	N/A	10	N/A
Barium	HQ = 1	6,500	N/A	0.5	N/A
Beryllium	Risk = 1E-4	180	N/A	0.4	N/A
Calcium	HQ = 1	--	N/A	10	N/A
Chromium	HQ = 1	460	N/A	0.5	N/A
Cobalt	HQ = 1	5,500	N/A	0.3	N/A
Copper	HQ = 1	3,400	N/A	100	N/A
Iron	HQ = 1	28,000	N/A	10	N/A
Magnesium	HQ = 1	--	N/A	5	N/A
Manganese	HQ = 1	13,000	N/A	0.5	N/A
Mercury	HQ = 1	28	N/A	0.5	N/A
Nickel	HQ = 1	1,800	N/A	0.5	N/A
Potassium	HQ = 1	--	N/A	50	N/A
Selenium	HQ = 1	460	N/A	0.5	N/A
Silver	HQ = 1	460	N/A	0.01	N/A
Sodium	HQ = 1	--	N/A	10	N/A
Thallium	HQ = 1	7.4	N/A	1	N/A
Vanadium	HQ = 1	830	N/A	0.5	N/A

a – Reported on a dry-weight basis.
N/A – Not applicable

Table B4.2.1.2: Average Levels in Unexposed Populations and Target Method Quantitation Limits for Biological Samples

Target Analyte	Units of Measure	Average Levels in Unexposed Populations	Method Quantitation Limit (MQL)
Biological Samples			
Blood-lead	µg/dL	10	1
Hair-arsenic	µg/g	10-15	2
Urine-arsenic	µg As /g Cr	50	0.05

Cr – Creatinine
Urine arsenic will be corrected for creatinine

B4.2.2 X-ray Fluorescence

XRF analysis will quantify arsenic, cadmium, lead and zinc concentrations for the fine fraction of the surface soil sample. Procedures for XRF analysis are outlined in the SOP located in Appendix A.

B4.2.3 Metals Analyses

B4.2.3.1 Confirmation Analyses

Confirmation metals analysis will be performed for twenty percent of samples analyzed by XRF. This analysis will serve to confirm results obtained by the primary laboratory. Confirmation analyses will be performed by an independent laboratory using either of the following instrumentation: ICP/AES or ICP/MS. Procedures for metals analysis are outlined in the respective EPA SW-846 analytical methods. Approved analytical method numbers are provided in Table B4.2.3.1.1 Either of these methods may be used providing the MQL for the parameter is achievable.

Table B4.2.3.1.1: Analytical Methods for Confirmation Analysis

Instrumentation	Parameter	SW-846 Method Reference
ICP	As, Pb, Cd, Zn	6010
ICP/MS	As, Pb, Cd, Zn	6020

ICP – Inductively Coupled Plasma
ICP/MS - Inductively Coupled Plasma/Mass Spectrometry

B4.2.3.2 PCOCs Metals Analyses

The subset of surface soil and soil core samples that have been identified for additional metals analysis (PCOCs Evaluation) must be analyzed on instrumentation that is capable of achieving the MQL requirements outlined in Section B4.2.1. Procedures for metals analysis are outlined in the respective EPA SW-846 analytical methods. Approved analytical method numbers are provided in Table B4.2.3.2.1

Table B4.2.3.2.1: Analytical Methods for PCOCs Evaluation

Instrumentation	Parameter	SW-846 Method Reference
ICP	All[a] except Hg	6010
ICP/MS	All[a] except Hg	6020
CVAA	Hg	SW-846 7470/7471
GFAA	As, Pb, Cd, Cu, Se, Tl	SW-846 7000 Series

a – All metals that can meet MQL requirements on that instrumentation.

B4.2.3.3 Environmental and Biological Sample Analyses

The following environmental and biologic samples: tap water, dust, blood lead, urine arsenic, hair arsenic, garden vegetables and surface soils co-located with garden vegetables will be analyzed using any of the methods outlined in Table B4.2.3.3.1.

Table B4.2.3.3.1: Analytical Method Requirements for Environmental and Biological Samples

Sample Medium	Parameter	Analytical Method
Tap Water	Pb	SW-846 6010, 6020 or 7421
Dust (House or Undisturbed)	As, Cd, Pb, Zn	XRF (SOP Appendix A)
Garden Vegetables	As, Cd, Pb, Zn	SW-846 6010, 6020
Co-located Surface Soils	As, Cd, Pb, Zn	SW-846 6010,6020
Blood	Pb	GFAA (SOP Appendix A)
Hair	As	Hydride Generation (SOP Appendix A)
Urine	As	Hydride Generation (SOP Appendix A)

GFAA – Graphite Furnace Atomic Absorption

B5 QUALITY CONTROL REQUIREMENTS

The principle objectives of any sampling and analysis program are to obtain accurate and representative environmental samples and to provide valid analytical data. The quality of data will be assessed through the use of QC samples performed on a regular basis. Laboratory QC samples will be analyzed as per analytical method protocols to evaluate whether laboratory procedures and analyses have been completed properly. For this project, the types of QC samples to be analyzed are defined and their role in the production of QC data are discussed in the following sections. Required QC samples are divided into sections as required by each analytical method.

In addition to the particular QC requirements identified in the subsequent sections, all analyses must be performed within holding times and must adhere to all procedures as outlined in the appropriate SOPs (Appendix A) or EPA SW-846 methods approved for this project.

B5.1 Analytical Methods

Quality control requirements for XRF and metals analyses are slightly different. Therefore, QC requirements for the two types of analyses are outlined separately.

B5.1.1 XRF Analysis

Method Blank: A method blank is composed of a matrix that is similar to investigative samples collected. For example, method blanks are composed of deionized water for water matrices and silica sand for solid matrices. Method blanks are analyzed to discern if laboratory-induced contamination is present during analysis at a frequency of at least 5% of samples analyzed (1 method blank per 20 samples analyzed or1 method blank per analytical batch, whichever is more frequent). Concentrations of target analytes must not exceed 1 x MDL.

Matrix Duplicate: A method duplicate is a sample that is split before preparation of the "XRF puck". The results of the two samples are compared to determine the precision observed. Method duplicates will be performed at a frequency of 5% (1 duplicate for every 20 investigative samples). The RPD for method duplicates is not to exceed 25%.

Laboratory Control Samples: A LCS originates in the laboratory or is provided as a standard reference material (SRM) by a manufacturer (eg. NIST) and contains target analytes of known concentration. Because LCSs are independent of the calibration standards, they are analyzed to verify the accuracy of the standards used to calibrate the instrument. At least one LCS must be analyzed in each analysis batch and analytical results must fall within manufacturer's limits.

B5.1.2 Metals Analysis

Method Blank: A method blank is composed of a matrix that is similar to investigative samples collected. For example, method blanks are composed of deionized water for water matrices and silica sand for solid matrices. Method blanks are analyzed to discern if laboratory-induced contamination is present during analysis at a frequency of 5% of samples analyzed (1 method blank per 20 samples analyzed or1 method blank per analytical batch, whichever is more frequent). Concentrations of target analytes must not exceed 1 x MDL.

Field Blank: A field blank is sample composed of a matrix that is similar to investigative samples collected and that is exposed to the field conditions in order to determine whether introduction of target analytes may be occurring during sampling. For example, field blanks will be collected for tap water and for house and undisturbed dust and will be composed of deionized water and the filter cartridge, respectively. Field blanks must be collected for appropriate matrices at a frequency of 5% of samples collected (1 field blank per 20 investigative samples collected). Concentrations of target analysis greater than 1 x MDL may suggest that field sampling-induced contamination of target analytes may have occurred.

Equipment Blank: An equipment blank is a liquid sample which is collected from during equipment decontamination. The final rinse (rinsate) is collected and analyzed to determine if equipment were adequately cleaned between sample locations. Concentrations of target analytes must not exceed 1 x MDL. Equipment blanks must be collected at a frequency of 5% (1 equipment blank per 20 equipment rinses).

Matrix Duplicate: A method duplicate is a sample that is split before digestion of the investigative sample. The results of the two samples are compared to determine the precision observed. Method duplicates will be performed at a frequency of 5% (1 duplicate for every 20 investigative samples). The RPD for method duplicates is not to exceed 25%.

Matrix Spike: The accuracy of an analytical method for a particular environmental sample matrix is evaluated by analyzing samples fortified with a known concentration of target analytes. A matrix spike is the analysis of a known concentration of target analytes added to an aliquot of the field sample. A matrix spike will be performed at a frequency of 5% (1 matrix spike for every 20 investigative samples). The percent recovery for the matrix spike is required to be 75-125%. If the matrix spike is outside of these acceptance limits and all potential error sources such as incorrect sample preparation or spiking concentrations, a post-digestion spike must be prepared and analyzed. The acceptance limits for the post-digestion spike is 85-115%.

Laboratory Control Samples: A LCS originates in the laboratory or is provided as a standard reference material (SRM) by a manufacturer (eg. NIST) and contains target analytes of known concentration. Because LCSs are independent of the calibration standards, they are analyzed to verify the accuracy of the standards used to calibrate the instrument. At least one LCS must be analyzed in each analysis batch and analytical results must fall within manufacturer's limits.

B6 INSTRUMENT/EQUIPMENT TESTING, INSPECTION AND MAINTENANCE REQUIREMENTS

Field equipment planned for use during this investigation is a portable XRF lead-paint analyzer. This instrument will be inspected daily to ensure it remains in good working condition. The instrument will be calibrated at the beginning of each day's use in accordance with the SOP (Appendix A). Calibrations must be acceptable before any measurements may be made. All information relating to the daily inspection, calibration and maintenance will be documented in the field logbooks.

B7 INSTRUMENT CALIBRATION FREQUENCY

Field and laboratory instrumentation, used for sample screening and analyses, will be calibrated in accordance with EPA guidance or the SOPs (Appendix A). Calibration procedures and frequencies are summarized in Appendix A. Traceable calibration standards will be obtained by the analytical laboratories. All documentation relating to

the receipt, preparation and use of standards will be recorded in the appropriate laboratory logbooks.

C ASSESSMENT AND OVERSIGHT

The following sections describe activities for assessing the effectiveness of the implementation of the project and associated quality assurance/quality control (QA/QC). The purpose of the assessment is to ensure that the SAP/QAPP is implemented as prescribed. The elements include assessments and response actions and reports to management as described in the following sections.

C1 ASSESSMENT AND RESPONSE ACTIONS

Assessment of laboratory analyses will be conducted through oversight of analytical procedures by ISSI, through optional laboratory audits conducted by ISSI and/or through submittal of performance evaluation samples. Laboratory audits will evaluate laboratory procedures to ensure that they follow GLP (Good Laboratory Practices) Guidelines and to ensure that they do not conflict with project requirements. If conflicts are noted, these must be addressed so that project requirements are met. Performance evaluation (PE) samples may be used as a tool for evaluating the accuracy of laboratory analyses. PE samples are standards submitted blind to the laboratory. The concentration is unknown to the laboratory analyzing the sample, but known to the submitter (ISSI). The laboratory reported results for the PE samples will be evaluated by comparison to the certified values provided to ISSI by the PE sample vendor. Acceptance criteria in terms of percent recovery windows may be established as appropriate to determine comparability. The degree of comparability expected between the certified values and the laboratory reported results will depend on a number of factors (which will be defined by ISSI) including the accuracy and precision reported by the vendor for the certified values and the comparability of the certification analysis method used by the vendor with the analysis methods used by the laboratory. The purpose of ISSI's oversight activities will be to document analytical procedures including changes, additions or deletions that occurred which were beyond the control of the analytical laboratory.

Two types of corrective actions may result from the laboratory oversight: immediate and long-term. Immediate corrective actions include correcting deficiencies or errors or correcting inadequate procedures. Long-term corrective actions are designed to eliminate the sources of deficiencies or errors. Corrective actions may be made through additional personnel training or procedural improvement.

D DATA VALIDATION AND USEABILITY

The following sections describe the requirements and methods for data review, validation and verification. In addition, the process for reconciling the data generated with the requirements of the data user is also defined.

D1 DATA REVIEW VALIDATION AND VERIFICATION

The process of data review, validation and verification is intended to provide consistent and defensible analytical results. Analytical data generated as part of this project will be reviewed and verified before they are incorporated into the project database. Full data validation will be completed on approximately 10 percent of the data generated for this project. Abbreviated validation will be completed on all succeeding analytical data. Abbreviated and full data validation criteria are described in Section D2.

D2 VALIDATION AND VERIFICATION METHODS

Data reporting consists of communicating summarized data in a final form. QA for reporting consists of measures intended to avoid or detect human error and to correct identified errors. Such methods include specification of standard reporting formats and contents of measures to reduce data transcription errors (Section A10).

D2.1 Validation

Full Validation: Full validation will be conducted on data packages for 10% of the samples analyzed for metals via XRF and independent metals analysis. This will be performed to ensure that data were produced with sufficient quality to establish confidence in the analytical results. The following elements will be reviewed for compliance as part of the full data validation:

- Method compliance
- Holding times
- Calibration
- Blanks
- Matrix spikes
- Method duplicates
- LCSs
- Other laboratory QC specified by the method
- Detection limits
- Analyte identification
- Analyte quantitation

Abbreviated Validation: Abbreviated validation will be completed on 100% of the analytical results for which full validation was not performed. This will be performed to ensure that data were produced with sufficient quality to establish confidence in the analytical results. The following elements will be reviewed for compliance as part of the abbreviated data validation:

- Method compliance
- Holding times
- Calibration
- Blanks
- LCSs
- Matrix spikes
- Method duplicates
- Other laboratory QC specified by the method

D2.2 Final Reporting

Laboratory Reports: All raw data and summary results of both data and summary statistics (means, standard deviations, ranges, etc.) will be provided by the laboratories. This information will be incorporated into ISSI's final report. Copies of the raw analytical data packages will be submitted to EPA for archival.

Study Report: A draft report of all the summary study design characteristics, sample analyses, data quality, correlation results and resulting analytical data shall be presented by the prime contractor (ISSI, Inc.). Simple statistical tests of group treatment differences will be performed and presented as discussed in Section A7. This report will undergo technical review by EPA. If necessary, comments to the draft report will be provided to ISSI and a final report will be issued.

D3 RECONCILIATION WITH DATA QUALITY OBJECTIVES

Information obtained from the Vasquez Boulevard and I-70 Residential Risk-based Sampling – Stage I Investigation will be evaluated through the data quality assessment (DQA) process to determine if the data obtained are of the correct quality and quantity to support their intended use. The DQA process consists of five steps as summarized below (USEPA 1996b).

Review the DQOs and Sampling Design: DQO outputs will be reviewed to ensure that they are still applicable. The sampling analysis and data collection documentation will also be reviewed for completeness and consistency with DQOs.

Conduct a Preliminary Data Review: Data validation reports will be reviewed to identify any limitations associated with the analytical data. Basic statistics will be utilized where applicable and meaningful graphs of the data will prepared as described in Section A7. This information will be used to learn about the structure of the data and to identify patterns, relationships or potential anomalies/outliers.

Select the Statistical Test: The most appropriate statistical procedure for summarizing and analyzing the data will be selected based on the review of the DQOs, the sampling design and the preliminary data review. Key underlying assumptions will be identified that must hold true for the statistical procedures to be valid.

Verify the Assumptions of the Statistical Test: The statistical test will be evaluated to determine whether the underlying assumption holds or whether departures from the assumptions are acceptable given the actual data or other information about the study.

Draw Conclusions from the Data: Calculations required for the statistical test will be completed and inferences drawn as a result of these calculations will be documented.

REFERENCES

USEPA. 1986. Test Methods for Evaluating Solid Waste, Physical/Chemical Methods SW-846, 3rd Ed. Office of Solid Waste and Emergency Response, Washington, DC.

USEPA. 1996a. Quality Management Plan for the U.S. Environmental Protection Agency, Region 8. September 1996.

USEPA. 1996b. Guidance for Data Quality Assessment. EPA QA/G-9. February 1996.

USEPA. 1998a. Sampling and Analysis Plan for North Denver Residential Soils. Prepared by URS Operating Services, Inc. March 1998.

USEPA. 1998b. Sampling Analysis Report for Removal Site Assessment. North Denver Residential Soils. July, 1998.

SBRC. 1997. In Vitro Method for Determination of Lead and Arsenic Bioaccessibility (SOP #1)

SBRC. 1997. Analysis for Lead and Arsenic in Extracts from Simplified In Vitro Bioavailability Procedure (SOP #2)

APPENDIX A: Extraction and Analytical Methods

APPENDIX B: Health-based Goals

APPENDIX A: Extraction and Analytical Methods

TECHNICAL STANDARD OPERATING PROCEDURE

--DRAFT--

SURFACE SOIL SAMPLING PROCEDURES

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for surface soil sampling to be used by employees of EPA Region VIII contractors/subcontractors supporting EPA Region VIII projects and tasks. This SOP describes the equipment and operations used for sampling surface soils in residential areas which will produce data that can be used to support risk evaluations. Site-specific deviations from the procedures outlined in this document must be approved by the EPA Region VIII Regional Project Manager, Regional Toxicologist, or On-Scene Coordinator prior to initiation of the sampling activity.

This SOP provides protocols for two different types of surface-soil sampling methods: discrete sampling and composite sampling. Depending on the data quality objectives outlined in the Project Plan, one of the following methods is appropriate.

2.0 RESPONSIBILITIES

Successful execution of the Project Plan requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role.

The Project Leader may be an EPA employee or contractor who is responsible for overseeing the surface soil sampling activities. The Project Leader is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the Project Leader to communicate with the Field Personnel specific collection objectives and anticipate situations that require any deviation from the Project Plan. It is also the responsibility of the Project Leader to communicate the need for any deviations from the Project Plan with the appropriate EPA Region VIII personnel (Regional Project Manager, Regional Toxicologist, or On-Scene Coordinator).

Field personnel performing soil sampling are responsible for adhering to the applicable tasks outlined in this procedure while collecting samples at residences. The field personnel should have limited discretion with regard to collection procedures, but should exercise judgment regarding the exact location of the Sample Point, within the boundaries outlined by the Project Leader.

3.0 EQUIPMENT

- Soil coring tool - Various makes of coring tools are acceptable and selection of the specific brand and make of tool should be specified in the Project Plan. Selection of the coring tool should be based on the individual characteristics of the soil to be sampled (e.g. clay, stony, soft etc.). At a minimum, the tool should be capable of retrieving a cylindrical plug of soil at least 3/4 inch in diameter and 3 inches long. A soil coring tool of this type is typically fabricated from stainless steel, has a hollow stem, is T-shaped and uses two handles to apply the force necessary for core collection. A plunger is used to press out the soil plug from the tip of the coring device. Plungers may be fitted with an adjustable stop to allow all but a given length of soil to be pushed from the coring tool. In all cases the procedures recommended by the manufacturers should be followed with regard to use of the coring tool. Coring tools with disposable plastic sleeves may be employed to minimize the decontamination effort.

- Collection containers - type to be specified in the Project Plan. Containers may be glass jars, plastic jars, or plastic bags.

- Scoop/spoon - for collecting surface soil samples. May be plastic or stainless steel. Must be lead free and unpainted.

- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.

- Field clothing and Personal Protective Equipment - as specified in the Project Plan.

- Squeeze bottle -for dispensing potable (drinking) quality water. Used to clean and decontaminate sampling equipment.

- Squeeze bottle - for dispensing deionized water. Used to clean and decontaminate sampling equipment.

- Wipes - disposable, paper. Used to clean and decontaminate sampling equipment.

- Field notebook -used to record progress of sampling effort and record any problems and field observations.

- Permanent marking pen - used to label sample containers.

- Sieves - if specified in the Project Plan. U.S. Standard # 10 (capable of passing material < 2 mm) and U.S. Standard # 60 (capable of passing material < 250 μm). Used to remove gravel and debris in the field to minimize shipping weight. Sieves mesh should be constructed of stainless steel or plastic and designed for soil processing.

- Measuring tape or pocket ruler -used to measure the length of soil core in the soil coring device.

- Plastic Buckets - used to receive rinse water generated in the course of tool cleaning, rinsing sieves, and used to collect the discarded soil from the coring tool.

- Trash Bag - used to dispose gloves and wipes.

- 0.01M HCl – used for equipment decontamination.

4.0 SAMPLING PATTERN

Discrete sampling requires soil collection from a single location and is used as a measure of the concentration at a single Sample Point. Composite sampling requires soil collection from multiple (sub-sample) points. These soils are then mixed and used as a measure of the concentration averaged over the entire area (zone).

The Project Plan will specify the pattern and order of sample collection. If compositing is to be done, the Project Plan will identify the areas and patterns used to group samples.

Care should be taken to avoid tracking soil from one area to another. As samples are taken sequentially, care should also be taken not to contaminate an area yet to be sampled with the residue of the sample that is currently being taken. In general one should move in a single direction through the sampling area. If an area is known or suspected of having a higher concentration of metals, all other considerations being equal, it should be sampled last to prevent cross contamination.

5.0 COLLECTION OF DISCRETE SURFACE SAMPLES USING A SOIL CORING DEVICE

A new pair of plastic gloves are to be worn at each Sample Point.

Locate the Sample Point on the ground specified by the Project Plan and clean the area free of twigs, leaves, and other vegetative material that can be easily be removed by hand. If the specified Sample Point is occupied by a rock, cobble or other hard objects of sufficient size that are incapable of easy removal by hand, move the Sample Point to the closest accessible location.

Place the soil coring tool on the ground and position it vertically. Holding the tool handle with both hands, apply pressure sufficient to drive the tool approximately 3 inches into the ground while applying a twisting force to the coring tool. Remove the tool by pulling up on the handle while simultaneously applying a twisting force. If the sample was retrieved successfully, a plug of soil approximately four inches long should have been removed with the coring tool.

If the Project Plan calls for coring of soil covered by turf-like vegetation (lawn), the coring tool should be pushed through the sod and the root mass extracted along with the soil core.

Hold the soil coring tool horizontally or place it on the ground. Place the coring tool plunger with the two inch stop inside the coring tool and push the soil plug out of the coring tool until the stop is encountered and two inches of soil remains inside. Using a clean spatula or knife, remove the soil collected at depth greater than two inches from the end of the sampling tool. Allow this soil to fall into the plastic bucket designated for excess soil material. Remove the stoppered plunger from the soil coring tool and using the unstoppered plunger, push the two-inch soil plug from the coring tool so that it falls directly into the sample container. Seal, label, and store the container as specified in the Project Plan.

Decontaminate the equipment as described in Section 12.0.

6.0 COLLECTION OF DISCRETE SOIL SAMPLES USING A SCOOP

A new pair of plastic gloves are to be worn at each Sample Point.

Locate the Sample Point on the ground specified by the Project Plan and clean the area free of twigs, leaves, and other vegetative material that can be easily be removed by hand. If the specified Sampe Point is occupied by a rock, cobble or other hard object of sufficient size to be incapable of easy removal by hand, move the Sample Point to a the closest accessible location.

Open a clean sample container. Using the metal spoon or scoop, excavate a hole in the soil approximately 2 inches in diameter and 2 inches deep while placing the excavated material directly inside the sample container. The sides of the excavated hole should be close to vertical to avoid sampling that is biased in favor of the upper layer of soil. Seal, label, and store the container as specified in the Project Plan.

Because decontamination procedures are time consuming, it may be desirable to use disposable spoons or have a quantity of scoops and spoons that may be used once and stored until the end of the day decontamination session.

7.0 COLLECTION OF COMPOSITE SAMPLES USING A CORING TOOL

A new pair of plastic gloves are to be worn in each Sampling Zone.

Locate the Sub-sample Point on the ground specified by the Project Plan and clean the area free of twigs, leaves, and other vegetative material that can be easily be removed by hand. If the specified Sub-sample Point is occupied by a rock, cobble or other hard object of sufficient size to be incapable of easy removal by hand, move the Sub-sample Point to a location closest to the original Sample Point.

Place the soil coring tool on the ground and position it vertically. Holding the tool handle with both hands, apply pressure sufficient to drive the tool approximately 3 inches into the ground while applying a slight twisting force to the coring tool. Remove the tool by pulling up on the handle while simultaneously applying a twisting force. If the sample was retrieved successfully, a plug of soil approximately three inches long should have been removed with the coring tool.

If the Project Plan calls for coring of soil covered by turf-like vegetation (lawn), the coring tool should be pushed through the sod and the root mass extracted along with the soil core.

Hold the soil coring tool horizontally or place it on the ground. Place the coring tool plunger with the two inch stop inside the coring tool and push the soil plug out of the coring tool until the stop is encountered and two inches of soil remains inside. Using a clean spatula or knife, remove the soil collected at depth greater than two inches from the end of the sampling tool. Allow this soil to fall into the plastic bucket designated for excess soil material. Remove the stoppered plunger from the soil coring tool and using the unstoppered plunger, push the two-inch soil plug from the coring tool so that it falls directly into the sample container. Repeat the steps outlined above until all the sub-samples from a given zone have been collected in the sample container.

Decontaminate equipment as described in Section 12.0.

8.0 COLLECTION OF COMPOSITE SAMPLES USING A SPOON OR SCOOP

A new pair of plastic gloves are to be worn in each Sampling Zone.

Locate the Sub-sample Point on the ground specified by the Project Plan and clean the area free of twigs, leaves, and other vegetative material that can be easily be removed by hand. If the specified Sub-sample Point is occupied by a rock, cobble or other hard object of sufficient size to be incapable of easy removal by hand, move the Sub-sample Point to a location closest to the original Sample Point.

Using the metal spoon or scoop, excavate a hole in the soil approximately 2 inches in diameter and 2 inches deep while placing the excavated material directly inside the compositing bowl. The sides of the excavated hole should be close to vertical to avoid sampling that is biased in favor of the upper layer of soil.

Repeat steps outlined above until all the sub-samples from a given zone have been collected in the sample container.

Decontaminate equipment as described in Section 12.0.

9.0 SITE CLEAN-UP

The Project Plan will address the methods used to fill holes generated by the sampling procedure. In general, it is desirable to fill sampling holes with clean, moist topsoil. The material should be poured into the hole and tamped down lightly.

Rinse water and 0.01M HCl, the unused fraction of soil cores, the roots of vegetation removed during sampling, and any unused soil generated in the course of sieving must be disposed of as specified in the Project Plan. Unless otherwise determined, this material should be regarded as hazardous waste and disposed accordingly.

10.0 RECORDING KEEPING AND QUALITY CONTROL

A field notebook should be maintained by each individual or team that is collecting samples as described in the Project Plan. The Project Plan will detail specific conditions which require attention, but at a minimum the following information should be collected.

This notebook information must include:

- date
- time
- personnel
- weather conditions
- a sketch of the sampling pattern that is filled in with sample identification numbers as the samples are collected
- locations of any samples and sub-samples that could not be acquired
- descriptions of any deviations to the Project Plan and the reason for the deviation.

Samples taken from soils with visible staining or other indications of non-homogeneous conditions should be noted. Draw a diagram that details the residence of each yard. Sample locations and sample numbers should be identified on the diagram. Superimpose a 5' x 5' grid onto the diagram and identify on the diagram sample locations (grid nodes) and sample numbers.

Field personnel will collect the proper type and quantity of quality control samples as prescribed in the Project Plan.

11.0 SAMPLE PREPARATION

Because data generated from collected surface soils will be used in evaluations of risk for metals exposure, sieving is required to obtain particle sizes that are the primary source of human exposure (< 250 μm). The soil sieving process produces a uniform material whose concentrations can be more accurately measured using laboratory techniques.

The option of whether to sieve soils prior to shipment to the laboratory as well as the location of sieving operations should be specified in the Project Plan. Soil sample must be dried and sieved in a controlled environment (laboratory) rather than in the field. Composite samples should have their sub-samples mixed prior to sieving.

11.1 Drying the Soils

Soils must be sufficiently dry prior to sieving. This may be determined by performing a "squeeze" test. The soil plug is pinched between a freshly gloved thumb and index finger. If the soil fragments and becomes powdery, the sample may be regarded as adequately dry for sieving. Alternatively, if soil squeezed in the palm of a freshly gloved hand becomes cohesive and retains its shape after squeezing, the soil has too much moisture for sieving.

If samples are not sufficiently dry, they should be air-dried by being allowed to stand in an open or partially covered sample container for 24 hours. Air-drying should be carried out in a warm room with moderate air circulation. If the soil is still too moist, it should be left to air dry for another 24 hours and tested again.

Rough guidelines for soil drying times are as follows:

- Sandy soil (24 hours)
- Silty soil (24 - 48 hours)
- Clayey soil (36 - 60 hours)

If samples are still not dry after these periods of air-drying or if drying times must be expedited, oven-drying may be necessary. Oven-dried samples will be dried to constant weight at 100 °C.

Once soil samples have been determined to be adequately dry, the sample plug or scoop should be manually crushed and broken up by squeezing the material with a freshly gloved hand. If the sample contains a section of grass sod, the soil should be shaken from the grass roots allowing this soil to mix with the other soil that will be sieved. The grass sod plug should be subjected to the screening process along with the other soil. Under no circumstances should the sample be ground (either against itself or against the compositing bowl or the sieving screens) as grinding generates particles that would not otherwise exist as part of the soil matrix.

11.2 Sieving

Sieving will be performed for each sample using clean equipment. Unprocessed soils (defined here as "raw soil") should first be sieved using a #10 screen, allowing particles <2 mm to pass through its mesh. Soils passing through a #10 screen will be defined here as "bulk soil". The bulk soil should then be sieved using a #60 screen, allowing particles <250 μm to pass through its mesh. Soils passing through a #60 screen are referred here as fine soil ("fines"). The screens may be stacked with the #10 screen on top and the #60 screen below. Covers (top and bottom) may be used as part of the sieving process if they are designed as part of the sieve set.

Sieving should be performed by pouring the soil sample on top of the sieve and shaking the screen rapidly back and fourth so that the material rolls over the screen mesh. The screen should occasionally be tapped against a hard surface to allow material to pass through mesh holes that have become clogged. Shaking should continue only as long as material above the screen contains particles smaller than the mesh opening. The screening process should not be used to break-up fragments of the soil core and materials should not be rubbed against the screen as a way of making them pass through the mesh.

The screens should be thoroughly cleaned prior each use. Decontamination procedures are described in Section 12.0.

12.0 DECONTAMINATION

Because decontamination procedures are time consuming, having a quantity of sampling tools sufficient to support decontamination at a maximum of once per day is recommended. All sampling and sieving equipment must be decontaminated prior to reuse.

The procedures to decontaminate all equipment is outlined below:

1) Remove visible soil.
2) Rinse equipment with potable water.
3) Rinse equipment with deionized water.
4) Rinse in a solution of 0.01M HCl.
5) Final rinse with deionized water.

Washing should be performed by sequential immersion of the equipment in buckets partially filled with these solutions. If necessary, a brush should be used to remove soil

material from screens and coring tools. Equipment should be set on clean toweling to dry. Equipment should be visibly dry before being used again.

Wipes, gloves, and rinse solutions must be disposed or stored properly as specified in the Project Plan.

13.0 GLOSSARY

Project Plan - The written document that spells out the detailed site-specific procedures to be followed by the Project Leader and the Field Personnel.

Sample Point - The actual location at which the sample is taken. The dimensions of a sample Point are 3/4" in diameter and 2" deep (core technique) or 2" across by 2" deep (spoon/scoop technique).

Discrete Sampling - A sample program in which material taken from a single Sample Point.

Composite Sampling - A sample program in which multiple Sample Points are compiled together and submitted for analysis as a single sample.

Sample zone - A unit of surface area subjected to a given sample program. A given zone usually is thought to contain similar metals concentrations or to be defined by a single set of exposure parameters.

Raw soils - Soil with sticks, leaves and debris removed but otherwise unprocessed.

Bulk soils - Raw soil that has passed through a U.S. Standard #10 sieve (< 2 mm).

Fine soil - Bulk soil that has passed through a U.S. Standard #60 sieve (< 250μm).

14.0 REFERENCES

USEPA, 1995. Residential Sampling for Lead: Protocols for Dust and Soil Sampling, Final Report, EPA 747-R-95-001, USEPA, March 1995, 38 p.

American Society for Testing and Materials, 1995. Standard Practice for Field Collection of Soil Samples for Lead Determination by Atomic Spectrometery Techniques, ASTM Designation: E 1727 - 95, October 1995, 3 p.

TECHNICAL STANDARD OPERATING PROCEDURE
XRF METALS ANALYSIS

--DRAFT--

1.0 General Discussion

1.1 Purpose of Procedure

This SOP is intended to describe the method for determining Pb, As, Cd, Cu, Fe, and Zn concentrations in bulk solid waste material using a KEVEX 0700/Delta XRF analyzer. This procedure is followed by all analysts at the Laboratory for Environmental and Geological Studies (LEGS).

1.2 Analysis Principal

Analyses of bulk powdered samples using the KEVEX 0700/Delta analyzer is based on the energy dispersive x-ray fluorescence of metal compounds within the sample. The emission of x-ray photons from the sample after excitation from selective monochromatic x-ray targets is integrated over time and ratio to the Compton peak to yield quantitative measurements.

The advantages of XRF include:

- High precision
- Large linear working range (7-10,000 ppm)
- Non Destructive
- Good Sensitivity (7-200 ppm)

The source of e-rays in the KEVEX system is a side window x-ray cube with a rhodium (Rh) anode. These x-rays may be focussed directly on the sample or used to excite secondary targets (Ti, Fe, Gd, Ag, Mo, or Ge) to produce optimum monochromatic x-rays. The x-rays are absorbed by the sample, exciting electrons to higher energy levels. As the electrons return to their ground state, photons characteristic to the elements found within the sample are emitted. The photons are detected by a solid-state Si-Li drifted detector. The energy values of these emitted photons are sorted by a multi-channel analyzer (MCA), producing a spectra of elements found in the sample. Spectra are collected over a specified time interval and stored as raw data files.

LEGS uses three different analysis conditions during a single sample tray run to maximize sensitivity for these elements (Table 1.2).

TABLE 1.2 Calculated detection limits for XRF.

Element	MDL_1	MDL_2
Pb	5 ppm	6 ppm
As	14 ppm	28 ppm
Cd	1 ppm	3 ppm
Cu	4 ppm	38 ppm
Zn	6 ppm	7 ppm
Fe	0.0006%	0.18%

MDL_1 = Detection limit calculated by taking (2 sigma) 2 times the standard deviation of ten consecutive analyses of Brazilian quartz

$MDL_2 = y0 + 2*z_0*S_y$ after method of McCormick and Roach, 1987. Using calibration curves.

2.0 Instrumentation and Forms

2.1 Instrumentation

The KEVEX 0700/Delta system consists of three main components: the x-ray cabinet, the computer console and a vacuum and coolant control system. The 0700 is equipped with a 60 Kv, 3.3 mA power supply for the Rh x-ray tube. The Si-Li detector is a 195 eV, 30 mm Quantum. Schematics of the x-ray analyzer and tube-target assembly are given in Figure 2.1. Additional information on components can be obtained from the KEVEX operations manual. Command structure is outlined in the KEVEX Quantex and Toolbox manuals.

2.2 Maintenance

Various maintenance procedures are performed on a routine bases to insure optimum instrument performance.

- Daily general cleaning of the work area to help control dust and avoid sample contamination.

- Daily check the MCA setting and insure it is set to "1".
- Weekly clean Bernoulli disk drives.
- Weekly check antifreeze coolant for x-ray tube.
- Monthly or as needed clean sample chamber, being careful not to damage detector thin window.
- Yearly replace vacuum pump oil.
- Yearly replace antifreeze coolant.
- Yearly replace filter on base of Bernoulli drives.

2.3 Forms

All sample batches are logged into the instrument notebooks, recording: project number, date, number of samples, number of elements, and client. Any system failures are also recorded in this notebook.

2.4 Supplies

The following supplies are kept on hand in order to insure minimal loss of time or analyses interruptions.

- Spare Iomega disk cleaning kit.
- Spare 40 MB Bernoulli disks.
- Sample cups, Chemplex #1530.
- Polypropelyne sheets, 2.5um thickness (Chemplex #100).
- Rotary pump oil.

- Reagent grade ethylene glycol.
- Printer paper.
- Printer ribbons.

3.0 Calibration Standards

Calibration is based on five levels of standards: a series of eight to eleven multi-element standards in an Al-Si matrix were developed at our lab; a flux monitor; a Brazilian Quartz blank; and NIST and/or USGS standards. When not in use all standards are stored in a laboratory safe.

All standards currently used at the laboratory are summarized in Table 3.1. Certificate of elemental concentrations when available are filed in the lab.

TABLE 3.1 Standards used in XRF Calibration.**

STANDARD	Pb ppm	As ppm	Cd ppm	Zn ppm	Cu ppm
SRM 2710	5532	626	22	6952	2950
SRM 2711	1162	105		350	114
USGS-GXR-1				740	1300
USGS-GXR-2				500	
USGS-GXR-3				220	
USGS-GXR-4				64	6500
USGS-GXR-5				50	360
USGS-GXR-6				120	105

** Included in our calibration are a series of standards made at the Laboratory for Environmental and Geological Studies. These standards are produced by mixing certified salts with a Al-Si matrix to give concentrations of 10, 30, 50, 100, 200, 500, 1000, 3000, 4000, 5000, and 10000 ppm. SRM 2710 and 2711 are NIST standards from Butte Montana.

TABLE 3.1 cont. Iron standards for XRF analyses.

STANDARDS	wt. % Fe (Total) **
USGS BHVO-1	12.2%
USGS RGM-1	1.87%
USGS STM-1	5.21%
USGS QLO-1	4.32%
USGS MAG-1	7.0%
USGS G-2	2.76%
USGS GXR-1	24.7%
USGS GXR-3	18.6%
USGS GXR-4	2.97%
USGS GXR-5	3.19%
NIST 120C	1.08%
NIST 697	20.0%
NIST 2710	3.38%
NIST 2711	2.29%

** Iron concentrations are not corrected for ZAF errors.

3.1 Use of Standards

Standard curves were developed for all elements by plotting instrument response (multiplied by flux monitor and ratio to Compton peak of target x-ray) in counts per second versus concentration in ppm. Current calibration curves are given in Figures 3.1-3.6. These curves represent a greater concentration range that is typically seen and a more general practice is to use a calibration curve that has a standard on either side of the highest and lowest unknown and is therefore "site specific".

The XRF system is recalibrated every three months using all standards unless internal QA standards indicate a drift of +/- 5% in calibration.

4.0 Procedure

Generally the KEVEX system is left running at all times and maintained at 20Kv and 0.2 Ma to extend x-ray tube life and improve tube stability. However, the computer system must be started and run through initial self-test and start-up. Matrix files are then recalled

from disk and tube ramp-up procedure run. Library space is checked and new disk is installed if inadequate space is left for sample storage.

4.1 Energy Calibration

Each week the MCA calibration is checked based on Al K alpha (1.45 KeV) and Cu (8.04 KeV) from a penny partially covered with aluminum foil. If accuracy of known peak localities is off by more than +/- 0.003 Kev the system is recalibrated using the internal EDV calibration routine.

4.2 Loading the Sample Chamber

The KEVEX system has a 16 position sample changer. On every sample run the zero and 1 position will contain the "monitor" and prep blank, while the 15th position will contain the mid-range internal standard. Following the described QA/QC (Table 5.1). The remaining positions will be loaded with sample unknowns which have been prepared following the sample preparation procedures 9.0.

Lab samples, standards, and QA/QC samples are loaded and their identification numbers are entered into the computers NAME file, then rechecked before saving.

4.3 Run Procedure

- Sample chamber loaded, closed and evacuated for a minimum of 15 minutes.
- While chamber is evacuating the first operating condition (Table 4.3) is recalled and set.

- Acquire procedure initiated to perform the following tasks:

  1. Acquire monitor
  2. Acquire spectra
  3. Remove escape and sum peaks
  4. Subtract background
  5. Deconvolute or integrate peaks
  6. Ratio peaks to target Compton peak
  7. Normalize to monitor value
  8. Store raw spectra
  9. Print results
  10. Move to next sample or start next condition
  11. Shut down analyzer

- Validation I (check sample numbers on print-out as samples are removed)

- Enter ratio values into current calibration curve on off-line PC.

- Validation II (check sample and ratio numbers in PC spreadsheet)

- Compile final sample and QA/QC results.

- Final report

- Validation III (check all sample numbers with chain of custody forms)

TABLE 4.3 OPERATING CONDITIONS

CONDITION	1	4	5
Kv	22	36	60
mA	1.5	1.5	1.5
TARGET	Ge	Ag	Gd
FILTER	0	0	0
ATMOSPHERE	VAC	VAC	VAC
ACQUISITION TIME	300 sec.	300 sec.	500 sec.
eV/Channel	10	20	40
Element	Zn + Cu + Fe	Pb + As	Cd
X-ray time	K alpha	Pb L Beta As K Beta	K alpha

5.0 Quality Assurance Quality Control

Analytical QA/QC procedures will follow methods and techniques consistent with EPA CLP where applicable and should ensure Level III quality data. The following procedures, Table 5.1, are followed routinely.

Table 5.1 QA/QC Procedures

Step	QC/QA	Frequency
Initial Calibration	Calibration Standards	As outlined in Calibration section
Continuing Calibration	Acceptance control limit will be values within +/- 25% RPD**.	Mid range standards run every 15 samples
Vacuum and tube checks	Monitor Standard	Every 15 samples
Precision	Duplicates 25% RPD* excepted value	Every 25 samples
Blanks	Lab blanks; 10ppm Pb, Cd; 20ppm As; 60ppm Cu; 40ppm Zn and 0.2% Fe are acceptance control limits.	Every 25 samples
NIST Standards	Traceability	Every 15 samples
CLP Checks	Intra-laboratory Check	Continuing process

Relative Percent Difference (RPD*)

$$= [(S1 - S2)/\{(S1 + S2)/2\}] * 100$$

S1 = initial value
S2 = value of duplicate

Relative Percent Difference (RPD**)

$$= [(R1-R0)/\{(R1+R0)/2\}] * 100$$

R1 = Value of mid internal standard
R2 = Known value for low internal standard

6.0 Corrective Actions

An essential part of a good QA/QC program is a well-defined set of procedures to be followed in the event that a problem develops either during data collection, reduction or compilation.

The corrective actions used are designed to identify problems early in the analyses and correct the problem in the most responsive manner. The laboratory Director is responsible for implementation of this program and will follow these steps:

1) Identify and define the problem

2) Determine a corrective action

3) Implement the corrective action

4) Verify that the corrective action resolved the problem

5) Document any corrective actions taken during the study

Corrective action taken during the XRF program will include three steps:

1) If flux monitor value is +/- 5% of accepted value of 1.0 then monitor is recollected prior to analyses.

2) If internal and NIST standards are outside of acceptable +/- 25% RPD then ALL preceding samples are reanalyzed

3) If NIST standards fail +/- 25% RPD repeatedly then system is recalibrated and all preceding samples are reanalyzed.

7.0 HEALTH AND SAFETY

Each individual operating the KEVEX x-ray fluorescence instrument will have read the "Radiation Handbook" prepared by the University (Quick Reference Guide and Table of Contents are supplied in Appendix A.) and follow all State guidelines for operation of x-ray equipment.

During preparation of sample cups researchers will wear latex gloves and particulate masks. All material that comes in contact with the samples or used to clean work surface areas will be placed in poly-bags for disposal.

8.0 FINAL REPORT

A final laboratory report will provide client with all XRF sample results, duplicates, blanks, SRMs and standard curves from this work plan. A hard copy as well as magnetic media will be provided.

9.0 SAMPLE PREPARATION

Generally samples are sent to our laboratory prepared by the client; however, if not, each sample will be strictly handled in the following manner. Splits for achieve can be made at any point depending on the clients wishes.

1) A sample is removed from shipping container and ID is verified on chain-of-custody form.

2) A new paper plate is marked with sample ID; sample and container are dispensed onto the plate. The plate is covered with a clean piece of computer paper and air-dried.

3) When dry, the sample is sieved using a 2 millimeter stainless steel sieve. If mud clods are a problem the technician will break these using only their fingers while wearing a clean set of disposable poly gloves.

4) The less than 2 mm sample is carefully placed back into the original sample container for further preparation. At this point sample ID on plate and container are cross checked. The coarser fraction is collected for later disposal. The sieve is cleaned with air and a poly brush, inspected, and ready for the next sample.

5) Approximately 30-50 grams of sample are then placed into a SPEX 8508 tungsten carbide grinding containers and pulverized for 1-2 minutes in a SPEX 8510 Shatterbox. If the sample size is greater than can be placed in a single tungsten carbide container a 30-50 gram split is used and the remaining sample archived.

6) This powdered sample is placed in new glass or poly vial using a clean piece of computer paper and labeled. Tungsten carbide containers are cleaned with water and air, then inspected. A sample preparation blank is prepared from a stock supply of silica sand following the same procedure every 25th sample.

7) The sample is thoroughly mixed within the vial by inverting and rotating the vial to ensure homogeneity and then approximately 5 grams are placed in a new CHEMPLEX 1530 cup and sealed. Labels on sample cups and vials are then cross-checked.

10.0 REFERENCES

KEVEX XRF TOOLBOX II Reference Manual P/N 7180-5060C, 1990.

KEVEX Delta XRF Analyst System Manual P/N 7180-0200, 1990.

McCormick, D. and Roach, A., 1987. Measurement, Statistics and Computation, John Wiley and Sons, London, pp. 508-513.

TAP WATER SAMPLING IN RESIDENCES FOR DETERMINATION OF RISK-BASED EXPOSURE TO LEAD

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standardized method to be employed at each residence for collection of tap water samples. This SOP describes the equipment and operations used for sampling residential interior water sources. The procedure outlines the method for sampling first flush and post-flush residential water that will ultimately be used for determination of lead concentrations.

2.0 SAMPLING PROCEDURE

2.1 Equipment

- 500 ml high density polyethylene sample bottle (small bottle)
- 1000 ml high density polyethylene sample bottle (large bottle) – the 750 ml mark is identified with permanent marker
- black permanent marker or ink pen
- 2 ampules (each containing 1 ml concentrated nitric acid - Reagent Grade)

2.2 Sample Location and Procedure

The following information or procedures will be followed or performed by the resident.

IMPORTANT! Collect water samples in the morning, prior to using any water sources in your home (ie: before flushing the toilet or running the sink). Both water samples will be collected from the main kitchen sink.

- Remove the cap from the small bottle and hold the bottle opening directly under the sink nozzle.
- Turn on the water and fill the small bottle to the top with the first draw of water from the kitchen sink. Turn off the water as soon as the sample bottle has been filled.
- Cap the small sample bottle tightly. Use a paper towel to dry the outside of the bottle.
- With a permanent marker or ink pen, write in the sampling time and date on the label.
- Turn on the water and let it run for 5 minutes before collecting the second sample.

- Remove the cap from the large bottle and hold the bottle opening directly under the sink nozzle.
- Fill the large sample bottle to the line (750 ml) on the outside of the bottle. Turn off the water after the sample bottle has been filled to the line.
- Cap the large sample bottle tightly. Use a paper towel to dry the outside of the bottle.
- With a permanent marker or ink pen, write in the sampling time and date on the label.
- Sample bottles do to need to be refrigerated prior to pick up.
- On the day of sampling, Environmental Protection Agency contractors will stop at each residence to pick up the sample bottles.

3.0 SAMPLE CONTRACTORS

The following information or procedures must be furnished or performed by the EPA contractors tasked with field sampling activities.

Before dropping off sample bottles at a residence:

- With a permanent marker or ink pen, label each sample bottle (500 ml and 1000 ml) with the sample identification number for each residence.
- On the outside of the 1000 ml sample bottle, draw a line with a permanent marker at the 750 ml mark.

After collecting sample bottles at a residence:

Immediately preserve water samples in the field by acidifying with nitric acid. This will reduce the tap water sample pH to less than 2.0.

- IMPORTANT! Put on latex gloves before opening nitric acid ampule.
- Gently break off the top of one nitric acid ampule and pour contents into the 500 ml bottle.
- Gently break off the top of one nitric acid ampule and pour contents into the 1000 ml bottle.
- Dispose of the ampules in a designated polyethylene container labeled "SHARPS ONLY".
- Remove gloves and place in a trash container.
- Note the time and date of sample collection and preservation in the field logbook.

4.0 REFERENCES

Bornschein. 1989. Midvale Community Lead Study, Appendix B: Quality Assurance Plan.

Designation: D 5438 – 93

AMERICAN SOCIETY FOR TESTING AND MATERIALS
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Standard Practice for Collection of Dust from Carpeted Floors for Chemical Analysis[1]

This standard is issued under the fixed designation D 5438; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This practice covers a procedure for the collection of a sample of carpet-embedded dust that can then be analyzed for lead, pesticides, or other chemical compounds and elements.

1.2 This practice is applicable to a variety of carpeted surfaces, such as home carpets, and has been tested for level loop and plush pile carpets specifically.

1.3 This practice is not intended for the collection and evaluation of carpet-embedded dust for the presence of asbestos fibers.

1.4 The values stated in SI units are to be regarded as the standard.

1.5 *This standard does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 422 Method for Particle-Size Analysis of Soils[2]

D 1356 Terminology Relating to Atmospheric Sampling and Analysis[3]

E 1 Specification for ASTM Thermometers[4]

E 337 Test Method for Measuring Humidity with a Psychrometer (The Measurement of Wet-Bulb and Dry-Bulb Temperatures)[3]

F 608 Laboratory Test Method for Evaluation of Carpet-Embedded Dirt Removal Effectiveness of Household Vacuum Cleaners[5]

3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D 1356.

3.1.1 *carpet-embedded dust*—soil and other particulate matter, approximately 5-μm equivalent aerodynamic diameter and larger, embedded in carpet pile and normally removable by household vacuum cleaners.

4. Summary of Practice

4.1 The sampling method described in this practice is taken from work published in Roberts, et al,[6,7] and Stamper, et al.[8]

4.2 Particulate matter is withdrawn from the carpet by means of a flowing air stream passing through a sampling nozzle at a specific velocity and flow rate and separated mechanically by a cyclone. The cyclone collects particles approximately 5-μm mean aerodynamic diameter and larger. The sampling system allows for height, air flow, and suction adjustments to reproduce systematically a specific air velocity for the removal of particulate matter from carpeted surfaces, so that these sampling conditions can be repeated.

4.3 The particulate matter in the air stream is collected in a catch bottle attached to the bottom of the collection cyclone. This catch bottle shall be capped for storage of the sample and transported to the laboratory for analysis.

5. Significance and Use

5.1 This practice may be used to collect embedded dust from carpeted surfaces for gravimetric or chemical analysis. The collected sample is substantially unmodified by the sampling procedure.

5.2 This practice provides for a reproducible dust removal rate from level loop and plush carpets and has the ability to achieve relatively constant removal efficiency at different loadings of surface dust.

5.3 This practice also provides for the efficient capture of semivolatile organic chemicals associated with the dust. The test system can be fitted with special canisters downstream of the cyclone for the capture of specific semivolatile organic chemicals that may volatilize from the dust particles during collection.

5.4 This practice does not describe procedures for evaluation of the safety of carpeted surfaces or the potential human exposure to carpet dust. It is the user's responsibility to evaluate the data collected by this practice and make such determinations in the light of other available information.

6. Interferences

6.1 There are no known interferences to the determina-

[1] This practice is under the jurisdiction of ASTM Committee D-22 on Sampling and Analysis of Atmospheres and is the direct responsibility of Subcommittee D22.05 on Indoor Air.

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[2] *Annual Book of ASTM Standards*, Vol 04.08.

[3] *Annual Book of ASTM Standards*, Vol 11.03.

[4] *Annual Book of ASTM Standards*, Vol 14.03.

[5] *Annual Book of ASTM Standards*, Vol 15.07.

[6] Roberts, J. W., Budd, W. T., Ruby, M. G., Stamper, V. R., Camann, D. E., Fortman, R. C., Sheldon, L. S., and Lewis, R. G., "A Small High Volume Surface Sampler HVS3 for Pesticides and Other Toxic Substances in House Dust," *Paper No. 91-150.2*, 84th Annual Meeting, Air & Waste Management Association, Vancouver, British Columbia, June 16-21, 1991.

[7] Roberts, J. W., and Ruby, M. G., "Development of a High Volume Surface Sampler for Pesticides," *U.S. Environmental Protection Agency Report No. EPA 600/4-88/036*, Research Triangle Park, NC, January 1989.

[8] Stamper, V. R., Roberts, J. W., and Ruby, M. G., "Development of a High Volume Small Surface Sampler for Pesticide and Toxics in House Dust," *Research Triangle Institute Report No. RTI/1111-01/01F*, Research Triangle Park, NC, June 1990. Included in supporting data, which are on file at ASTM Headquarters. Request RR:D22-1010.

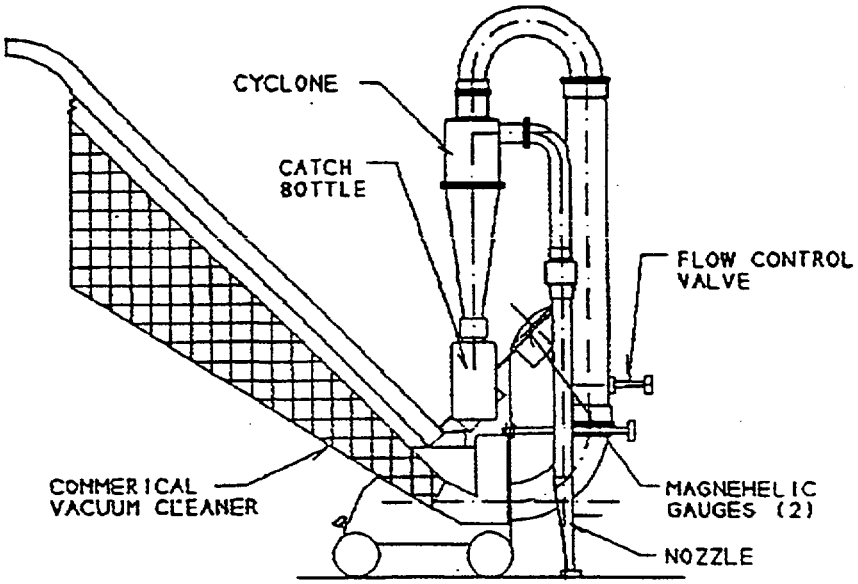

FIG. 1 Carpet Dust Sampler Using a Commercial Vacuum Cleaner as the Suction Source

tion of dust loadings covered by this practice.

7. Apparatus

7.1 *Sampling Apparatus*, which may be acquired commercially[9] (as shown in Fig. 1) or constructed as follows:

7.1.1 The dimensions of the sampling apparatus (nozzle size, cyclone diameter, cyclone inlet diameter, etc.) are interdependent. The flow rate must produce a sufficient velocity both at the carpeted surface and in the cyclone. The cyclone must have a cut diameter of 5 µm at the same velocity that will provide a horizontal velocity of 40 cm/s at 10 mm from the nozzle in the carpet material. The fundamental principles of this device have been discussed in detail in Roberts, et al.[6,7]

7.1.2 *Nozzle*—The edges and corners of the sampling nozzle shall be rounded to prevent catching the carpet material. The nozzle must be constructed to allow for sufficient suction to separate loose particles from the carpet and carry them to the cyclone. It must have an adjustment mechanism to establish the nozzle lip parallel to the surface and to achieve the proper suction velocity and pressure drop across the nozzle. A nozzle 12.4 cm long and 1 cm wide, with a 13-mm flange and tapered to the nozzle tubing at no more than 30 deg, will yield the appropriate velocities when operated as specified in Section 11.

7.1.3 *Gaskets*—Gaskets in joints should be of a material appropriate to avoid sample contamination.

7.1.4 *Cyclone*—The cyclone shall be of a specific size such that a given air flow allows for separation of the particles 5-µm mean aerodynamic diameter and larger. The cyclone must be made of aluminum or stainless steel, and the catch bottle must be made of clear glass or fluorinated ethylene propylene (FEP) to avoid contamination and allow the operator to see the sample.

7.1.5 *Flow Control System*—The flow control system shall allow for substantial volume adjustment. The suction source must be capable of drawing 12 L/s (265 CFM) through the system with no restrictions other than the nozzle, cyclone, and flow control system connected. A commercial vacuum cleaner[10] can be used for this purpose.

7.1.6 *Flow Measuring and Suction Gages*—The use of Magnehelic[11] gages for measurement of the pressure drop at the nozzle and for control of the flow rate for the entire system is considered adequate and applicable for this sampling practice.

7.2 *Other Equipment*:

7.2.1 *Stopwatch.*

7.2.2 *Masking Tape and Marking Pen*, for outlining sections for sampling.

7.2.3 *Clean Aluminum Foil and Clean Glass or FEP Jars*, for the collection and storage of samples.

7.2.4 *Thermometer* (see Specification E1).

7.2.5 *Relative Humidity Meter* (see Test Method E 337).

7.2.6 *Shaker Sieve*, as specified in Method D 422, with 100 mesh-screen above the pan to separate the fine dust below 150 µm.

7.2.7 *Analytical Balance*, sensitive to at least 0.1 mg and having a weighing range from 0.1 mg to 1000 g.

[9] Available from CS$_3$, Inc., P.O. Box 5186, Bend, OR 97708.

[10] The Model 7100Z Royal, available from Royal Appliance Manufacturing Co., Cleveland, OH 44143, or equivalent, has been found to be suitable for this purpose.

[11] Registered trademark of Dwyer Instruments, Inc., Michigan City, IN 46360.

D 5438

SAMPLE DATA SHEET

Operator _____ Date _____ Sample Ident. #: ____

Sampling site _____

Type of Carpet: Plush ___ Level Loop ___ Multilevel ___ Shag ___
Type of Vacuum: Upright ___ Canister ___ Other _____

Last Vacuumed _____ Temp. _____ Humidity _____%

Comments: _____

Location of Area Sampled: _____ Area _____m^2

Sketch of Area Sampled:

Leak Check: Yes__ No__; 20 second cleaning @ end: Yes __ No __

Total Sample Time:__minutes __seconds Flow ΔP ____ Nozzle ΔP ___

Bottle final Wt: _____g Tare Wt: _____g Net Wt: _____g

Pan & Sample Wt: _____g Pan Tare Wt: _____g Net Wt: _____g

Total Dust: _____ grams/m^2
Fine Dust: _____ grams/m^2

Cyclone Sample #: _____

Lab Sample #: _____

FIG. 2 Sample Data Sheet for Sampling for Carpet Dust

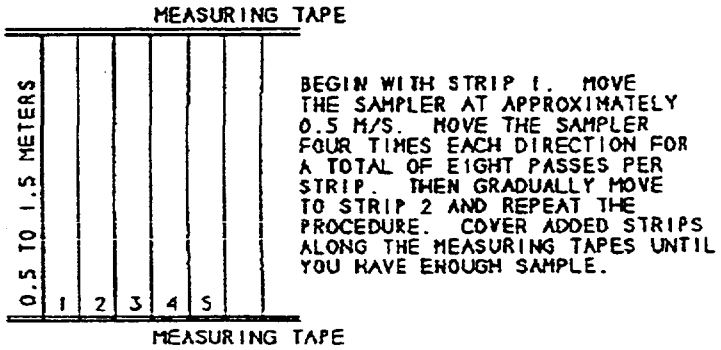

FIG. 3 Example of a Typical Sampling Procedure

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[12]

8.2 Methanol is required for sampling train cleaning after sample collection.

9. Sampling Strategy

9.1 The overall sampling strategy should be designed to address the goals of the study. Users should consider factors such as foot traffic volume, types of activities, proximity to potential sources, etc. The sampling strategy should be described in the sampling report so it can be taken into consideration when readers are comparing loadings or concentrations, or both, to those obtained from other studies. The ideal sampling location(s) for the beginning of the test procedure are an area that conforms with the protocol for the user's overall sampling strategy. For example, when sampling in a home for child exposure assessment, protocol may require the selection of a carpeted area for sampling where small children play or are likely to play.

10. Pretest Preparation and Calibration

10.1 *Calibration*—The sampling system described in this practice does not have any calibrated flow devices other than the cyclone and the Magnehelic gages. The cyclone used for the separation of the particles must be designed to give proper separation at varying flow rates throughout the sampling range of the system. The pressure gages and any other devices (that is, temperature gage) used for testing purposes should be calibrated against a primary standard.

10.1.1 *Pressure Gages*—Pressure gages shall be calibrated against an inclined manometer or other primary standard prior to any field test. One means of checking a Magnehelic gage is to set a flow rate through the sampling system with a manometer and then switch to the Magnehelic gage. If the difference in the readings is more than 3 %, the gage is leaking or is in need of repair or calibration. This should be done at two different flow rates when checking the gage.

10.1.2 The cyclone flow measurement is calibrated with a laminar flow element, spirometer, or roots meter. See the appendix for cyclone calibration with a laminar flow element.

10.2 *Pretest Preparation:*

10.2.1 Each catch bottle to be used shall be clean and inspected for any contamination. The bottles should be marked with masking tape and a marking pen for identification of the test site, time, and date.

10.2.2 The sampling train shall be inspected to ensure that it has been cleaned and assembled properly.

10.2.3 The sampling train shall be leak-checked prior to sampling. This can be accomplished by placing a mailing envelope or a piece of cardboard beneath the nozzle and switching on the suction source. The flow Magnehelic gage should read 5 Pa (0.02 in. H_2O) or less to ensure that the system is leak free. If any leakage is detected, the system shall be inspected for the cause and corrected before use.

11. Sampling

11.1 *Pre-Test Survey*—Immediately prior to testing, complete a data form recording all requested information and sketch the area to be sampled. (See Fig. 2 for a sample data form.)

11.2 Select a sampling area according to the established protocol for your sampling campaign. This should be determined prior to testing.

11.3 A typical sampling procedure may use measuring tapes placed on the carpet so that they are parallel to each other and on either side of the portion of carpet to be sampled (Fig. 3). The measuring tapes should be between 0.5 and 1.5-m apart and extended as far as practical. They should be taped to the carpet with masking tape every 30 cm.

11.4 Place the sampler in one corner of the sampling area and adjust the flow rate and pressure drop according to the type of carpet (see 11.8). The two factors that affect the efficiency of the sampling system are the flow rate and pressure drop at the nozzle. The pressure drop at the nozzle is a function of the flow rate and distance between the surface and the nozzle flange.

11.5 Clean the wheels and nozzle lip with a clean labora-

[12] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, NY, and the "United States Pharmacopeia."

tory tissue[13] immediately before sampling. Begin sampling by moving the nozzle between the ends of the two measuring tapes. The sampler is then moved back and forth four times on the first strip, moving the sampler at approximately 0.5 m/s. (The widths of the strips are defined by the width of the sampling nozzle.) Effective nozzle width is 13 cm for the CS_3 sampler. Move in a straight line between the numbers on the measuring tape. Angle over to the second strip on the next pass gradually, and repeat four double passes. After sampling approximately 0.5 m^2, determine the amount of collected material in the bottom of the catch bottle. As a rough estimate, the collection of dust to a depth of 6 mm (0.25 in.) in a 55-mm diameter catch bottle corresponds to approximately 6 to 8 g. If there is less than 6 mm of dust, sample an additional 0.5 m^2 next to the area already sampled. Hair, carpet fibers, and other large objects should be excluded from the sample when estimating the quantity collected.

11.6 Continue sampling in the area laid out until an adequate sample is collected. Switch off the vacuum. The catch bottle can now be removed, labeled, and capped for storage and analysis. Record the dimensions of the sampled area on the data sheet.

11.7 If the rug area to be sampled is very dirty, or has not been cleaned frequently, care must be taken to avoid filling up the cyclone catch bottle on the first sample area. If it is suspected that this will be the case, start with a 0.25-m^2 sampling area. Then take a second and a third area as before, until the catch bottle is 75 % full.

11.8 Adjust the flow rate and nozzle pressure drop to values that approximate those given in Table 1. Use the same flow rate and pressure drop on multilevel and shag carpets as that used for plush carpets.

12. Sample Analysis

12.1 After collection of the sample in the catch bottle, the sample may be left in the same bottle or transferred to another container for transport to the laboratory. The procedure for sample handling is different for metals and organic chemicals. Samples for organic analysis should be maintained at 4°C to the extent possible. (Samples should not be frozen before sieving, as this could alter the particle size distribution.) Storage at ambient temperature is appropriate for samples that will be analyzed only for metals, but cooling the sample is also acceptable.

12.2 If the sample will be analyzed for pesticides or other organic chemicals, transfer the dust from the cyclone catch bottle onto the middle of a piece of aluminum foil that has been cleaned by washing with pesticide-free methanol or hexane. Fold the foil into a small package carefully, keeping the dust in the middle. Place the foil pouch in a clean glass jar. Cover the jar opening with another piece of precleaned foil and secure the lid to the jar. Seal the seam of the lid to the jar with polytetrafluoroethylene tape. Place the sample jar in an ice chest to keep it cool during transport to the laboratory. Label the jar for reference.

12.3 If the sample will be analyzed for metals, it can be transferred from the catch bottle to a new polyethylene "zipper" seal sample bag.[14] Seal the zipper, and tape the seal with any marking tape that will adhere well to the polyethylene bag. Label the sample for reference.

12.4 Sieve the samples for 5 min in a shaker in accordance with Method D 422, with a 100-mesh screen above the pan, to determine the weight of fine dust below 150-μm mean diameter.

12.5 Alternative methods for the storage, shipment, and preparation of samples for analysis may be required for some analytes and should be prescribed for specific sampling protocols. The FEP catch-bottle may be used for storage and shipping.

TABLE 1 Approximate Values for Flow Rate and Nozzle Pressure Drop

Carpet Type	Flow Rate	Nozzle Pressure Drop
Plush	9.5 L/s (20 CFM)	2.2 kPa (9 in. H_2O)
Level loop	7.5 L/s (16 CFM)	2.5 kPa (10 in. H_2O)

13. Sampler Cleaning

13.1 After the sample bottle is removed, open the flow control valve to maximum flow, tip the sampler back so that the nozzle is approximately 5 cm (2 in.) off the floor, and switch the vacuum on. Place a hand covered by a rubber glove over the bottom of the cyclone and alternate closing and opening the cyclone for 10 s to free any loose material adhering to the walls of the cyclone and tubing. It is not necessary to catch this small amount of dust, as it is usually much less than 1 % of the collected sample.

13.2 Remove the sampler to a well-ventilated cleaning area free of dust. Remove the cyclone and elbow at the top of nozzle tubing from the sampler. Use a 50-cm long by 3-cm diameter (20 by 1.25-in.) brush to clean the nozzle, and clean all related items up to and including the cyclone and catch bottle with reagent grade methanol. This wash can be analyzed at the discretion of the operator. The total amount of dust removed in the air and wet cleaning is usually much less than 1 % of the collected dust. The air and wet cleaning is performed to prevent contamination from passing from one sample to another.

14. Data Analysis

14.1 Weigh the sieved dust sample with an analytical balance accurate to 0.1 mg.

14.2 Calculate the dust weight by subtracting the weight of the pan sample from the final weight according to Method D 422.

14.3 Calculate the loading for dust per square metre (g/m^2) by dividing the final dust weight by the area sampled (expressed in m^2).

14.4 When the analysis results are received from the laboratory, it is possible to calculate the loading of lead, pesticides, or other analytes per square metre of carpet area ($\mu g/m^2$) in the same way.

14.5 The concentration of any element or chemical associated with the dust may be determined by analysis.

[13] Kimwipes, available from Kimberly-Clarke Corp., Roswell, GA 30076, or equivalent.

[14] Fisherbrand Trademark, available from Fisher Supply, 711 Forbes Ave., Pittsburgh, PA 15219, or equivalent.

TABLE 2 Sampling Efficiency Using Modified Laboratory Test Method F 608[A]

Parameters	Carpet Type	
	Plush	Level Loop
Flow rate (L/s)	8.4	7.6
Delta P (kPa)[B]	2.3	2.5
Mean % of mass collected in cyclone	69.5	66.8
Standard deviation	1.2	2.8
Number of tests	8	3

[A] Carpet dust loading was 15.9 g/m².
[B] Pressure drop at nozzle.

15. Precision and Bias[15]

15.1 Tests for dust collection efficiency have been performed using Laboratory Test Method F 608 modified by passing it through a 100-mesh sieve.[6,7] The results are given in Table 2.

15.2 Tests performed with a fine particle filter downstream of the cyclone showed that 99 % or more of the collected test dust was retained in the cyclone catch bottle.[6,7]

15.3 Tests performed as in 15.2, but with test dust containing lead, showed that 99 % or more of the lead collected was retained in the cyclone catch bottle.[6,7]

15.4 Tests performed as in 15.2, but with test dust fortified with pesticides, showed that 97 % or more of the pesticides collected were retained in the cyclone catch bottle. The pesticides tested were chlordane, aldrin, chlorpyrifos, heptachlor, and diazinon.

15.5 Tests were conducted on conditioned carpets, as described in Laboratory Test Method F 608.

[15] Supporting data have been filed at ASTM Headquarters. Request RR:D22-1010.

16. Keywords

16.1 carpet; cyclone; dust; floors; metals; organic chemicals; particle size; particulate matter; vacuum

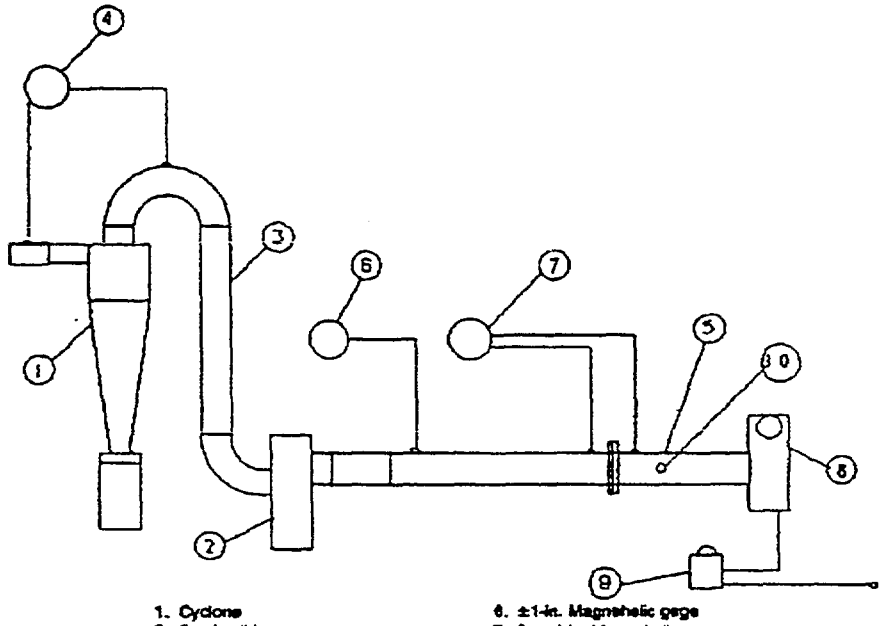

FIG. X1.1 Calibration Using a Laminar Flow Element

APPENDIX

(Nonmandatory Information)

X1. CALIBRATION OF CYCLONE USING A LAMINAR-FLOW ELEMENT

X1.1 Assemble the necessary components (see Fig. X1.1).

X1.1.1 *Cyclone.*

X1.1.2 *Suction/Blower.*

X1.1.3 *Flow Control Valve,* 1 to 2.5 kPa (0 to 10 in.).

X1.1.4 *Magnehelic Gage,* 1 to 2.5 kPa (0 to 10 in.)

X1.1.5 *Laminar Flow Element* (with manufacturer's certified calibration), with pressure gages and dial thermometer.

X1.1.6 *Suction/Blower,* with power transformer; leak check the system by plugging the inlet to the cyclone and observing the pressure gage.

X1.1.7 Activate Blowers 2 and 8.

X1.1.8 Open the flow control valve on Flow Control Valve 3 so that 2.0 kPa (8.0 in. H_2O) registers on Pressure Gage 4. Then adjust Variable Autotransformer 9 so that 0.0 kPa (0.0 in. H_2O) registers on Pressure Gage 6. Some adjusting of the flow control valve will be necessary.

X1.1.9 Check Pressure Gage 7 for the gas flow reading and record the flow.

X1.1.10 Adjust the flow through the cyclone to 2.5 kPa (10.0 in. H_2O), and repeat the procedure. This action should provide a gas flow rate through the cyclone. This should be between 7.1 and 8.5 L/s (15 to 18 CFM).

UNDISTURBED DUST SAMPLING AT RESIDENCES FOR DETERMINATION OF RISK-BASED EXPOSURE TO METALS

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standardized method for interior surface dust sampling for undisturbed locations within a residence. These protocols will be employed by employees of EPA Region VIII or contractors and subcontractors supporting Region VIII projects and tasks. This SOP describes the equipment and operations used for exposure-based sampling of residential interior surface dust where ongoing metal contaminants exposure is suspected. The procedure outlines the method for vacuum sampling of interior surface dust that will ultimately be used for evaluation of health risk. Site-specific deviations from the procedures outlines in this document must be approved by the EPA Region VIII Remedial Project Manager or Regional Toxicologist prior to initiation of the sampling activity.

2.0 RESPONSIBILITIES

Individuals obtaining metals concentration measurements are responsible for performing the applicable tasks outlined in this procedure when conducting dust sampling at residences. The Project Leader may be an EPA employee or contractor who is responsible for overseeing the dust sampling activities. The Project Leader is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the Project Leader to communicate with the Field Personnel specific collection objectives and anticipate situations that require deviation from the Project Plan. It is also the responsibility of the Project Leader to communicate the need for any deviations from the Project Plan with the appropriate EPA Region VIII personnel (Regional Project Manager or Regional Toxicologist).

3.0 PROTOCOL FOR COLLECTION OF SETTLED DUST SAMPLES FOR METALS DETERMINATION USING VACUUM SAMPLING

This protocol provides for the collection of settled, undisturbed dust samples from surfaces using a low-volume vacuum method. The protocol is suitable for the collection of settled dust samples from both hard or smooth and highly textured surfaces, such as brickwork and rough concrete, and soft, fibrous surfaces, such as upholstery and carpeting. Note that these samples are specifically intended to be collected in attics that

are not use as living space. These samples serve to provide historical estimates of dust sources. Information on dust loading will not be quantified.

In brief, this protocol involves placing a template at the area or location to be sampled, and then vacuuming the area inside the template in a standardized pattern of vertical and horizontal passes with the collection nozzle. Because the area of the template is known, the results of the sampling effort can be expressed either as concentration (µg metal per gram of dust) or as dust loading (µg of metal per m^2).

Note: The total mass of dust collected in the composite sample should be at least one gram, since this is sufficient to support either an XRF analysis or a wet chemistry analysis. If a 1-gram sample is not collected, several "sub-locations" may be sampled until sufficient mass is collected. A sub-location is defined as an area within the same sampling location (i.e. child's room).

3.1 Equipment

- Air sampling pump – A portable, battery-powered air pump that is capable of maintaining a flow rate of 3.0 L/min through a filter cassette. The inlet of the pump must be fitted with a nipple to accept the tubing sized to fit tightly on the outlet side of a filter cassette.
- Air monitoring cassette – (with a 0.8 micron polycellulose acetate filter)
- Disposable shoe covers (optional)
- Secondary sample collection container – resealable plastic bags for holding and transporting the filter cassettes.
- Plastic tubing – plastic, flexible tubing sized to fit tightly on both the inlet and outlet of a filter cassette and the inlet of the air-sampling pump.
- Collection nozzle – A piece of stainless steel or carbon-impregnated plastic machined or molded on each end as follows: one machined or molded end to accept the tubing sized to fit tightly on the inlet side of a filter cassette; the other machined or molded to form a thin rectangular opening of ½" by $^3/_{64}$".
- Calibrated rotameter – equipped with inlet and outlet fittings sized to fit tubing used to connect the filter cassette to the air-sampling pump. This is used to calibrate the flow rate of the personal air pump.
- Stop Watch - Digital watch with elapsed time feature for calibration of air pump. Not necessary is air flow meter has electronic time sensor.

3.2 Sample Location

Samples will be collected in uninhabited attic spaces.

3.3 Sampling Procedure

3.3.1 Create a floor plan of the uninhabited area to be sampled. Select surface to be sampled. Do not walk on or touch the surface to be sampled. Include a drawing of the floor plan in your field logbook. The unique sample location identifiers should be included on the drawing.

On floors with hard surfaces, dust will often migrate to the edges of a room. Therefore, the most likely place to find an adequate supply of surface dust might be an area immediately adjacent to a wall.

3.3.2 Connect the calibrated monitoring pump to the air monitoring cassette with tubing.

3.3.3 Connect the air monitoring cassette to the collection attachment with tubing.

3.3.4 Holding the vacuum in sampling position above the template, turn on the monitoring pump and ensure the flow rate is at 3.0 ± 0.2 liters per minute.

3.3.5 Vacuum visible dust in the undisturbed location. Vacuum as many different sub-locations within the area as possible, paying particular attention to corners of the room and edges of attic beams.

3.3.6 Remove the dust cassette from the collection attachment and replace the end plugs. Ensure that the end plugs are secured for shipment. If necessary, secure the plugs with duct tape.

3.3.7 Label the dust cassette with sufficient information to uniquely identify the sample. This should include at a minimum, the sample ID number and collection date. See section 5.1.3 for other labeling recommendations.

3.3.8 All measurements should be recorded in a field notebook, so that area calculations can be repeated at a later time if needed. Discard any gloves worn during sampling and any sampling debris into a trash bag. Remove the trash bag when leaving the dwelling. If the template is a reusable type, clean the template with several clean wipes.

4.0 DECONTAMINATION PROCEDURES

4.1 Tubing: Because tubing is not very expensive and decontamination procedures can be time consuming, tubing should be discarded after each use and be replaced with clean, unused tubing for each new sample.

5.0 QUALITY CONTROL

Adherence to quality assurance/quality control (QA/QC) procedures is an important part of field sample collection. QA/QC procedures, including documentation requirements, field QC samples, reference material check samples, and contamination avoidance are presented in this section.

5.1 Documentation

All field data related to sample collection must be documented. A field notebook or sample log form can be used to record field collection data. It is recommended to use both types of documentation records (field notebooks and preprinted sample log forms) for assuring collection of all relevant field data. Field data entries on document records must adhere to the following requirements:

5.1.1 General Documentation Requirements

- All entries must be made using indelible ink.
- Each page (notebook or form) must include the name of the person making the entries and the data of entries found on the page.
- Any entry errors must be corrected by using only a single line through the incorrect entry (no scratch outs) and marked with the initials of the person making the correction and the date of correction.
- An initial page that correlates initials to a specific name must be generated and maintained with field data records to trace any initials used in notebooks and on data forms.

5.1.2 Specific Sampling Site Documentation Requirements

- General sampling site description.
- Project or client name, address, and city/state location.
- Information as to what specific collection protocol was used (e.g., SOP #).
- Information as to the use of interim storage and sample shipment mechanisms.
- Prefield weight data including stabilization conditions for filter cassette or wipes.
- Postfield weight data including stabilization conditions for filter cassette or wipes.

5.1.3 Documentation Required for Each Sample Collected

- An individual and unique sample identifier and date of collection. This must be recorded on the sample container in addition to the field data records (notebook or form).
- Name of person collecting the sample and specific sampling location data from which the sample was removed.

5.2 QC Samples

- Field blanks. Field blank samples are used to identify any potential systematic metals contamination present in the filter cassette or wipes and handling of samples during field collection and laboratory analysis activities. Field blanks should be periodically collected throughout the sampling day at each sampling site. Each field blank must be labeled with its own unique identifier. The identifier for all blanks should appear similar to other field samples to mask the identity of the blank from the laboratory (i.e., blanks are submitted to the laboratory in a blind manner). It is recommended that field blanks be collected at a frequency of field samples (1 field blank per 20 field samples).

  Filter Cassette: Field blanks should be collected in the same manner used to collect field samples with the exception that the vacuum sampling nozzle is pointed away from the floor and air is drawn through the filter cassette. Each cassette designated as a field blank should be removed from the plastic bag, inlet and outlet caps pulled off, the tubing and sampling nozzle attached, and then this procedure is reversed. The vacuum pump is turned on.

- Blind Reference Material Samples. Reference materials should be periodically submitted to the laboratory for analysis as a check on adherence to proper laboratory sample preparation and instrumental analysis methods.

  Filter cassette: Prepare a blind reference material by placing an accurately weighed portion (1.0 ± 0.1g) of a reference material into a pre-weighed glass vessel. Vacuum the material into the filter cassette from the vessel. Reweigh the vessel to determine how much reference material was vacuumed into the filter. Document all measurements. Two blind reference material samples should be prepared for every 20 field samples. These should have arsenic and lead concentrations at 1x and 3x the respective action level. For example: the 1x sample should have arsenic

and lead concentrations of 70ppm and 500ppm respectively, whereas the 3x sample would have arsenic and lead concentrations of 210ppm and 1500ppm. The recommended frequency of these QC samples to be submitted to the laboratory for metals determination be at least 10% of field samples (2 reference material per 20 field samples).

5.3 Contamination Avoidance

The following work practices should be followed to prevent cross-contamination of samples:

- Avoid disturbing and tracking dust from one location to another by:
  - identifying and clearly marking all sampling locations upon arrival at the sampling site,
  - avoiding walking through or over any of the marked sampling location areas, and
  - instructing field teams members to pull on new disposable shoe covers upon each entry into the building (this is especially significant if field teams have been walking through known exterior contamination sources).
- Use a new pair of powderless gloves at each sampling location.
- Inspect all sampling equipment for cleanliness prior to collection of each sample. Always clean suspect equipment if in doubt.
- Do not open sample collection containers until needed to collect each sample.
- Immediately remove and dispose of shoe covers and gloves when sampling is complete.

6.0 REFERENCES

Bornschein. 1989. Midvale Community Lead Study, Appendix B: Quality Assurance Plan.

EPA. 1995a. Residential Sampling for Lead: Protocols for Dust and Soil Sampling. Final Report. U.S. Environmental Protection Agency, Office of Pollution Prevention and Toxics. EPA 747-R-95-001.

EPA. 1995b. Sampling House Dust for Lead. Basic Concepts and Literature Review. U.S. Environmental Protection Agency, Office of Pollution Prevention and Toxic Substances. EPA 747-R-95-007.

EPA. 1996. Sampling Manual for IEUBK Model. Prepared by Roy F. Weston. Document control number 4800-045-0019.

LEAD PAINT SCREENING AT RESIDENCES FOR DETERMINATION OF RISK-BASED EXPOSURE TO LEAD

1.0 PURPOSE

The purpose of this standard operating procedure (SOP) is to provide a standardized method to be employed by employees of EPA Region VIII or contractors and subcontractors supporting Region VIII projects and tasks. This SOP describes the equipment and operations used for screening residential interior and exterior paint. The procedure outlines the method for screening for lead paint that will ultimately be used for risk-based determination of lead concentrations. Site-specific deviations from the procedures presented in this document must be approved by a Project Manager or Regional Toxicologist prior to initiation of the sampling activity.

2.0 RESPONSIBILITIES

Individuals obtaining lead concentration measurements are responsible for performing the applicable tasks outlined in this procedure when conducting lead paint screening at residences. The Project Leader may be an EPA employee or contractor who is responsible for overseeing the lead paint screening activities. The Project Leader is also responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the Project Leader to communicate with the Field Personnel specific screening objectives and anticipate situations that require deviation from the Project Plan. It is also the responsibility of the Project Leader to communicate the need for any deviations from the Project Plan with the appropriate EPA Region VIII personnel (Regional Project Manager or Regional Toxicologist).

3.0 PROCEDURE

3.1 Equipment

- Lead paint analyzer (portable X-ray fluorescence detector)
- Calibration standards for lead paint analyzer
- Field logbook
- Indelible ink pen (black or blue)

3.2 Interior Screening Location

Two painted surfaces (woodwork and walls), in three separate rooms of the residence will be screened. Unpainted surfaces (eg: paneling, wallpaper, and unpainted woodwork) will not be screened. Additionally, surfaces that are treated with stain rather than paint will not be screened.

The walls and three rooms of the residence will be screened. These areas will be the living room or family room (whichever room has the television), the kitchen, and the subject child's bedroom. The subject child is defined as the child living at the residence that is supplying a blood lead sample. If no child is present in the home, an adult bedroom should be screened. If these rooms are unpainted, then other alternative rooms will be selected. Alternative rooms should be selected based upon the relative frequency that it is occupied by the family. Note all sampling locations on a diagram of the entire residence (see Section 4.0).

In order to characterize the paint and surfaces in a given room, we will screen at least one painted wall and one painted trim in the room. When screening the woodwork, three separate readings will be taken at three different locations on the woodwork. Similarly, three separate measurements will be taken on painted walls within a room. One reading will be taken on each of three separate wall areas, either on the same wall or on different walls within a room. If all walls are painted the same color, then the three readings can be taken from one wall. If the walls are painted different colors, then a reading from each of the different colored walls should be included. The locations and reported results of all lead paint XRF readings shall be marked in the field logbook.

3.3 Exterior Screening Location

Three separate areas on the outside of the residence structure should be screened for lead. As with the interior screening, unpainted or stained surfaces should not be screened. The selection of areas to be screened should be based upon: (1) differences in the color and/or age of paint, (2) condition of the paint (ie: flaking areas vs. good condition paint), (3) differences in surfaces (ie: painted walls vs. trim). The locations and reported results of all lead paint XRF readings shall be marked in the field logbook.

3.4 Instrument Calibration

Follow instructions provided by the manufacturer for calibration of the lead paint analyzer. It is necessary to calibrate the lead analyzer prior to taking actual readings in the residence. Two standards should be used to calibrate the lead paint analyzer. One standard should be low (at or near the instrument detection limit) and another should be a lead standard that falls at the middle of the linear range of the instrument. Triplicate measurements of each standard must be made

and documented in the field logbook. Calibration measurements must fall within 90-110% of the known value. If any measurement exceeds these acceptance limits, the analyst must uncover and correct the problem. Calibration criteria must be met prior to analysis of any investigative samples.

3.5 Sampling Procedure

- Place the lead paint analyzer on the designated surface and open the shutter. Flat surfaces provide more accurate readings than curved surfaces, therefore curved surfaces will be avoided.
- The lead content of the paint will appear as a visual numeric display on the XRF. NOTE: Begin taking readings with the XRF analyzer set to display low lead readings, if needed, set the XRF to display high lead readings.
- Read the number displayed on the XRF and record each reading in the field logbook.
- For exterior surfaces, evaluate the condition of the painted exterior surfaces using a rating scale of 1-3 (1 = paint is intact and adhering completely to the surface; 2 = some deterioration at the surface with slight paint flaking; 3 = extremely deteriorated surface with flaking paint and loose paint adhesion to the surface) and record this information in the field logbook.

4.0 DOCUMENTATION

- Draw a diagram of the residence in the field logbook. Mark each reading location on the diagram. Annotate each reading so that the numerical measurement is known at each location. For example: the first reading = 1, the second reading = 2, etc.)
- Mark all lead paint readings for both the calibration and screening surfaces in the field logbook. Identify each reading as displayed on the lead paint analyzer. Include the corresponding measurement number as identified on the diagram.

5.0 REFERENCES

Bornschein. 1989. Midvale Community Lead Study, Appendix B: Quality Assurance Plan.

Standard Operating Procedure for Sampling Garden Vegetables at Lead Contaminated Sites

1.0 PURPOSE

The purpose of this Standard Operating Procedure (SOP) is to provide a standardized method for sampling garden vegetables to be used by employees of EPA Region VIII, contractors, and subcontractors supporting EPA Region VIII projects and tasks. This SOP describes the equipment and operations used for sampling surface soils of residential. Site-specific deviations from the procedures outlined in this document must be approved by the Project Leader at EPA Region VIII prior to initiation of the sampling activity.

This SOP provides protocols for sampling three different types of garden produce: leafy vegetables, root vegetables, and garden fruits. Each type of vegetable requires a different method of sample collection.

Depending on the data quality objectives outlined in the Project Plan, one of the following methods is appropriate.

2.0 RESPONSIBILITIES

Successful execution of the Project Plan requires a clear hierarchy of assigned roles with different sets of responsibilities associated with each role.

Field personnel obtaining lead concentration measurements are responsible for performing the applicable tasks outlined in this procedure while collecting samples at residences. The field personnel should have limited discretion with regard to collection procedures but, within the boundaries outlined by the Project Leader, exercise judgment regarding the exact location of the Sampling Point.

The Project Leader is responsible for checking all work performed and verifying that the work satisfies the specific tasks outlined by this SOP and the Project Plan. It is the responsibility of the Project Leader to communicate with the Field Personnel specific collection objectives and anticipate situations that require any deviation from the Project Plan. It is also the responsibility of the Project Leader to communicate the need for any departures from the Sampling Plan with EPA region VIII.

3.0 EQUIPMENT

- Collection containers - type to be specified in the Project Plan. Containers may be glass jars, plastic jars, or plastic bags.
- Shovel - for collecting root vegetables. Must be lead free and unpainted.
- Knife - for cutting leafy vegetables and garden fruit from plants.
- Clippers - for cutting samples from plants.
- Gloves - for personal protection and to prevent cross-contamination of samples. May be plastic or latex. Disposable, powderless.
- Field clothing and Personal Protective Equipment - as specified in the Project Plan.
- Squeeze bottle -for dispensing potable (drinking) quality water. Used to clean and decontaminate sampling equipment.
- Squeeze bottle - for dispensing de-ionized water. Used to clean and decontaminate sampling equipment.
- Wipes - disposable, paper. Used to clean and decontaminate sampling equipment.
- Field notebook -used to record progress of sampling effort and record any problems and field observations.
- Permanent marking pen - used to label sample containers.
- Plastic Buckets - used to receive rinse water generated in the course of tool cleaning.
- Trash Bag - used to dispose gloves and wipes.
- Vegetable Brush -used to scrub soil from root vegetables.
- Laboratory Surfactant - used to decontaminate sampling equipment. Alconox is a brand in common use.

4.0 SAMPLING PATTERN

The Sampling Plan will specify the pattern and order of sample collection. If compositing is to be done, the Sampling Plan will identify the areas and patterns used to group samples.

5.0 COLLECTION OF LEAFY VEGETABLES

The Sampling Plan will specify the type and quantity of leafy vegetables that are to be collected. A clean pair of gloves should be worn at all times and changed between samples of each type of plant (leafy vegetable, root, or garden fruit) within each garden or sampling zone. Harvest the plant as it would be prepared in a commercial garden. In the case of lettuce, cut the entire plant at ground level. In the case of beet greens, if the root is not to be analyzed, the leaves may be cut from the plant leaving the beet root in the ground. Using a clean plastic bucket that has been partially filled with deionized water, immerse the sampled plant material in deionized water and wash the dust and soil from the plant using gentle agitation for one minute. Drain the water from the plant and place the entire head in a plastic ziploc bag that has been properly labeled. Place the sample immediately in a secured cooler at a temperature of 4^{o} C.

After all leafy vegetables within a given garden have been sampled and before these same tools are used to sample another garden, the sampling tools and samples should be scraped and rinsed free of visible soil and thoroughly washed with a laboratory surfactant cleaner (Alconox). After washing tools should be dried with wipes or allowed to air dry.

6.0 COLLECTION OF ROOT VEGETABLES

The Sampling Plan will specify the type and quantity of root vegetables that are to be collected. A clean pair of gloves should be worn at all times and changed between samples of each type of plant (leafy vegetable, root, or garden fruit) within each garden or sampling zone. Harvest the plant as it would be prepared in a commercial garden. Carrots will be used as an example, but the same procedure should be used for any sub-surface vegetable. For carrots, use the spade to dig around the individual carrot (or multiple adjacent carrots if called for in the Sampling Plan) to be sampled. Bring up the entire shovel-full of earth and carrots. Shake the carrots free from the earth mass. If the Sampling Plan calls for it, the green tops of the carrots should be removed before washing. The carrots should be washed by repeatedly immersing them in a bucket of deionized water and scrubbing the taproot surface with a brush. Change the water in the bucket if it becomes extremely muddy. The carrots should have no visible soil adhering to the surface of the taproot. After scrubbing, the carrots should be given a final rinse in a

bucket of deionized water, placed in a labeled plastic bag, and stored in a secure cooler at 4° C.

Garden soil excavated as part of root vegetable sampling should be replaced and the surface lightly tamped down. Vegetable material not to be analyzed but generated as part of the sampling effort (carrot greens for example) should be disposed .

After all root vegetables within a given garden have been sampled and before these same tools are used to sample another garden, the sampling tools and samples should be scraped and rinsed free of visible soil and thoroughly washed with a laboratory surfactant cleaner (Alconox). After washing tools should be dried with wipes or allowed to air dry.

7.0 COLLECTION OF GARDEN FRUITS

The Sampling Plan will specify the type and quantity of garden fruit that is to be collected. A clean pair of gloves should be worn at all times and changed between samples of each type of plant (leafy vegetable, root, or garden fruit) within each garden or sampling zone. Harvest the plant as it would be prepared in a commercial garden. In the case of tomatoes, the fruit may be pulled directly from the vine using a gloved hand. It may be ncessary to use decontaminated clippers to cut the tomato fruit from the plant. Rinse the tomatoes by immersing them in a bucket partially filled with deionized water and wipe them while under water to remove any visible soil. Place the fruits in a new appropriately labeled ziploc bag and store in a cooler at 4° C.

After all root vegetables within a given garden have been sampled and before these same tools are used to sample another garden, the sampling tools and samples should be scraped and rinsed free of visible soil and thoroughly washed with a laboratory surfactant cleaner (Alconox). After washing tools should be dried with wipes or allowed to air dry.

8.0 RECORDING KEEPING AND QUALITY CONTROL

A field notebook should be maintained by each individual or team that is collecting samples. The Sampling Plan will detail specific conditions which require attention, but at a minimum the following information should be collected. This notebook information must include but is not restricted to date, time, personnel, field weather conditions, a sketch of the garden layout including the proximity of any buildings and visible paint chips or soil staining. The goals for note taking should be twofold:

1.) The field records should be sufficient for a second team of field personnel to return to the same location and obtain results substantially like the first.

2.) Anomalous values found in the course of laboratory analysis may be traced back to a specific location or procedure and the cause of the value understood.

As specified in the Sampling Plan, at various points in the sampling effort field personnel should expect to collect quality control samples. These may include filling two sample containers with soils that have been taken from the same core or surface excavation. It may also include filling two sample containers with homogeneous bulk or fine soil material.

11.0 DECONTAMINATION

At the end of each day's sample collection work, all sampling, tools must have visible soil removed, be washed with drinking water and rinsed with dionized water. Wipes, gloves, and rinse solutions must be disposed or stored properly as specified in the Sampling Plan.

1.0 OBJECTIVES

The objectives of this Standard Operating Procedure (SOP) are to specify the proper methodologies and protocols to be used during metal speciation of various solid samples (including tailings, slags, sediments, dross, bag house dusts, and paint), residential soils and dusts for metals. The metal speciation data generated from this SOP may be used to assess the solid samples as each phase relates to risk. Parameters to be characterized during the speciation analyses include particle size, associations, stoichiometry, frequency of occurrence of metal-bearing forms and relative mass of metal-bearing forms. This electron microprobe (EMP) technique, instrument operation protocols and sample preparation to be used during implementation of the Metals Speciation SOP are discussed in the following sections.

2.0 BACKGROUND

To date, numerous metal-bearing forms of soils have been identified from various environments within western mining districts (Table 2-1) (Emmons et al., 1927; Drexler, 1991 per. comm.; Drexler, 1992; Davis et al., 1993; Ruby et al., 1994; CDM, 1994; WESTON, 1995). This listing does not preclude the identification of other metal-bearing forms, but only serves as an initial point of reference. Many of these forms are minerals with varying metal concentrations (e.g., lead phosphate, iron-lead oxide, and slag). Since limited thermodynamic information is available for many of these phases and equilibrium conditions are rarely found in soil environments, the identity of the mineral class (e.g., lead phosphate) will be sufficient and exact stoichiometry is not necessary.

It may be important to know the particle-size distribution of metal-bearing forms in order to assess potential risk. It is believed that particles less than 250 microns (μm) are most available for human ingestion and/or inhalation (Bornschein, et al., 1987). For this study, the largest dimension of any one metal-bearing form will be measured and the frequency of occurrence weighted by that dimension. Although not routinely performed, particle area can be determined. It has been shown (CDM, 1994) that data collected on particle area produces similar results. These measurements add a considerable amount of time to the procedure and limit the total number of particles or samples that can be observed in a study.

Mineral association may have profound effects on metal the ability for solubilization. For example, if a lead-bearing form in one sample is predominantly found within quartz grains while in another sample it is free in the sample matrix, the two samples are likely to pose significantly different risk levels to human health. Therefore, associations of concern

include the following:

1) free or liberated
2) inclusions within a second phase
3) cementing
4) alteration rims

3.0 SAMPLE SELECTION

Samples should be selected and handled according to SOP #6 of this project manual.

4.0 SCHEDULE

A schedule for completion of projects performed under this Metals Speciation SOP will be provided in writing or verbally to the contractor along with monthly reporting requirements if large projects are performed. These schedules are based on an aggressive analytical program designed to ensure that the metals speciation analyses are completed in a timely period. Monthly reports are expected to reflect schedule status.

5.0 INSTRUMENTATION

Speciation analyses will be conducted at the Laboratory for Environmental and Geological Studies at the University of Colorado, Boulder or other comparable facilities. Primary equipment used for this work will include:

Electron Microprobe (JEOL 8600) equipped with four wavelength spectrometers, energy dispersive spectrometer (EDS), BEI detector and the TN-5600 data processing system. RJ Lee ZEPPELIN and DATALINK hardware may be used for image storage and processing. An LEDC spectrometer crystal for carbon and LDE-1 crystal for oxygen analyses will be used.

6.0 PRECISION AND ACCURACY

The precision of the EMP speciation will be evaluated based on sample duplicates analyzed at a frequency of 10%. The accuracy of the analyses will be estimated based on a number of methods, depending on the source of the data. Data generated by the "EMP point count" will be evaluated statistically based on the methods of Mosimann (1965) at

the 95% confidence level on the frequency data following Equation 1.

$$E_{0.95} = 2P(100-P)/N \qquad \text{(Eq. 1)}$$

Where: $E_{0.95}$ = Probable error at the 95% confidence level

P = Percentage of N of an individual metal-bearing phase based on percent length frequency

N = Total number of metal-bearing grains counted

It is very important that every attempt be make to ensure that a minimum of 100-200 total particles are counted in order to provide a statistically meaningful particle count. If the contractor specifies, either the NIST 2710 or 2711 "Montana soils" can be speciated for traceability.

Quantitative elemental analysis, primarily performed on slag or other variable, metal-bearing forms, will have precision and accuracy evaluated on counting statistics and reproducibility of NIST or other certified standards using conventional EMP methods. In general, site-specific concentrations for these variable, metal-bearing forms will be determined by performing "peak counts" on the appropriate wavelength spectrometer. Average concentrations will then be used for further calculations. Data on specific gravity will be collected from referenced databases or estimated based on similar compounds.

7.0 PERSONNEL RESPONSIBILITY

The analysts will carefully read this SOP prior to any sample examination.

It is the responsibility of the laboratory supervisor and designates to ensure that these procedures are followed, to examine quality assurance (QA) and replicate standards, and to check EDS and WDS calibrations. The laboratory supervisor will collect results, ensure they are in proper format, and deliver them to the contractor.

Monthly reports summarizing all progress, with a list of samples speciated to date with data analyses sheets (DAS), will be submitted each month.

It is also the responsibility of the laboratory supervisor to notify the contractor representative of any problems encountered in the sample analysis process.

8.0 METHODOLOGY

9.0 Sample Preparation

Grain mounts, 1.5 inches in diameter, of each sample will be prepared using air-cured epoxy. The grain mounting is performed as follows:

1) Log the samples of which polished mounts will be prepared.

2) Inspect all disposable plastic cups, making sure each is clean and dry.

3) Label each "mold" with its corresponding sample number.

4) All samples will be split to produce a homogeneous 1-4 gram sample.

5) Mix epoxy resin and hardener according to manufacturer's directions.

6) Pour 1 gram of sample into mold. Double check to make sure sample numbers on mold and sample match. Pour epoxy into mold to just cover sample grains.

7) Use a new wood stirring stick with each sample, carefully blend epoxy and grains so as to coat all grains with epoxy.

8) Set molds to cure at ROOM TEMPERATURE in a clean restricted area. Add labels with sample numbers and cover with more epoxy resin. Leave to cure completely at room temperature.

9) One at a time remove each sample from its mold and grind flat the back side of the mount.

10) Use 600 grit wet abrasive paper stretched across a grinding wheel to remove the bottom layer and expose as many mineral grains as possible. Follow with 1000 grit paper.

11) Polish with 15 um oil-based diamond paste on a polishing paper fixed to a lap. Use of paper instead of cloth minimizes relief.

12) Next use 6um diamond polish on a similar lap.

13) Finally polish the sample with 1um oil-based diamond paste on polishing paper, followed by 0.05 um alumina in water suspension. The quality should be checked after each step. Typical polishing times are 30 minutes for 15 um, 20 minutes for 6 um, 15 minutes for 1 um, and 10 minutes for 0.05 um.

    NOTE: use low speed on the polishing laps to avoid "plucking" of sample grains.

14) Samples should be completely cleaned in an ultrasonic cleaner with isopropyl alcohol or similar solvent to remove oil and finger prints.

15) To ensure that no particles of any metal are being cross-contaminated during sample preparation procedures, a blank (epoxy only) mold will be made every 20th sample (5% of samples) following all of the above procedures. This mold will then be speciated along with the other samples.

16) Each sample must be carbon coated. Once coated the samples should be stored in a clean dry environment with the carbon surface protected from scratches or handling.

8.2 Point Counting

Counts are make by traversing each sample from left-to-right and top-to-bottom as illustrated in Figure 8-1. The amount of vertical movement for each traverse would depend on magnification and CRT (cathode-ray tube) size. This movement should be minimized so that NO portion of the sample is missed when the end of a traverse is reached. Two magnification settings generally are used. One ranging from 40-100X and a second from 300-600X. The last setting will allow one to find the smallest identifiable (1-2 micron) phases.

The portion of the sample examined in the second pass, under the higher magnification, will depend on the time available, the number of metal-bearing particles, and the complexity of metal mineralogy. A maximum of 8 hours will be spent per sample.

8.3 Data Presentation

Analysts will record data as they are acquired from each sample using the LEGS software, which places all data in a spreadsheet file format. Columns have been established for numbering the metal-bearing phase particles, their identity, size of longest dimension in microns, along with their association (L = liberated, C= cementing, R = rimming, I = included). The analyst may also summarize his/her observations in the formatted data summary files.

The frequency of occurrence and relative metal mass of each metal-bearing form as it is distributed in each sample will be depicted graphically as a frequency bar-graph. The particle size distribution of metal-bearing forms will be depicted in a histogram. Size-histograms of each metal-bearing form can be constructed from data in the file.

Data from EMP will be summarized using two methods. The first method is the determination of FREQUENCY OF OCCURRENCE. This is calculated by summing the longest dimension of all the metal-bearing phases observed and then dividing each phase by the total.

Equation 2 will serve as an example to the calculation.

$$F_M \text{ in phase-1} = \frac{\Sigma (PLD)_{\text{phase 1}}}{\Sigma (PLD)_{\text{phase-1}} + \Sigma (PLD)_{\text{phase-2}} + \Sigma (PLD)_{\text{phase-n}}} \quad \text{(Eq. 2)}$$

Where:

F_M = Frequency of occurrence of metal in a single phase.
PLD = An individual particle's longest dimension
$\%F_M$ in phase-1 = F_M in phase-1 * 100

These data thus illustrate which metal-bearing phase(s) are the most commonly observed in the sample or relative volume percent.

The second calculation used in this report is the determination of RELATIVE METAL MASS. These data are calculated by substituting the PLD term in the equation above with the value of M_M. This Term is calculated as defined below.

$$M_M = FM * SG * ppm_M \quad \text{(Eq. 3)}$$

Where:

M_M = Mass of metal in a phase
SG = Specific Gravity of a phase
ppm_M = Concentration in ppm of metal in a phase

The advantage in reviewing the RELATIVE METAL MASS determination is that it gives one information as to which metal-bearing phase(s) in a sample are likely to control the total bulk concentration for a metal of interest. For example, PHASE-1may by relative volume comprise 98% of the sample; however, it has a low specific gravity and contains only 1,000 parts per million (ppm) arsenic. PHASE-2 comprised 2% of the sample, has a high specific gravity, and contains 850,000 ppm of arsenic. In this example it is PHASE-2 that is the dominant source of arsenic to the sample.

Finally, a concentration for each phase is calculated. This quantifies the concentration of each metal-bearing phase. This term is calculated as defined below (Eq. 4).

$$ppm_M = M_M * \text{Bulk metal concentration in ppm} \quad \text{(Eq. 4)}$$

8.4 Analytical Procedure

A brief visual examination of each sample will be made, prior to EMP examination. This examination may help the operator by noting the occurrence of slag and/or organic matter. Standard operating conditions for quantitative and qualitative analyses of metal-bearing forms are given in Table 8-1. Quality control will be maintained by analyzing standards and duplicates at regular intervals (see next section).

The backscattered electron images will be examined using two settings: one for light-element matrices (slag or organic) and the second for heavy-element matrices (lead sulfide or lead carbonate etc.). This procedure will minimize the possibility that metal-bearing minerals may be overlooked during the scanning of the polished grain mount. The scanning will be done manually in a manner similar to that depicted in Figure 8-1. Typically, the magnification used for scanning all samples except for airborne samples will be 40-100X and 300-600X. The last setting will allow the smallest identifiable (1-2 um) phases to be found. Once a candidate particle is identified, then the backscatter image will be optimized to discriminate any different phases that may be making up the particle or defining its association. Identification of the metal-bearing phases will be done using both EDS and WDS on a EMP, with spectrometers peaked at sulfur, oxygen, carbon and the metal of concern (M). The size of each metal-bearing phase will be determined by measuring in microns the longest dimension. A maximum of 8 hours will be spent in scanning and analyzing each mount.

Quantitative Analyses

Quantitative analyses are required to establish the average metal content of the metal-bearing minerals, which have variable metal contents as: Iron-(M) sulfate, Iron-(M) oxide, Manganese-(M) oxide, organic, and slag. These determinations are important, especially in the case of slag, which is expected to have considerable variation in their dissolved metal content. Results will be analyzed statistically to establish mean values. They may also be depicted as histograms to show the range of metal concentrations measured as well as the presence of one or more populations in terms of metal content. In the later case, non-parametric statistics may have to be used or the median value has to be established.

Associations

The association of the metal-bearing forms will be established from the backscattered electron images. Particular attention will be paid in establishing whether the grains are totally enclosed, encapsulated or liberated. The rinds of metal-bearing grains will be identified. Representative photomicrographs of backscatter electron images establishing the association of the principal metal-bearing forms will be obtained for illustration purposes. A positive/negative, black and white film (Polaroid 55) will be used or a 128x128 (minimum) binary image in ".tif" format may be stored. Recorded on each photomicrograph and negative will be a scale bar, magnification, sample identification and phase identification. Abbreviations for the identified phases should be used. Examples are listed in Table 8-2. A final list must be submitted with the laboratory report.

8.5 Instrument Calibration and Standardization

The WDS will have spectrometers calibrated for the metal of concern, carbon, oxygen and sulfur on the appropriate crystals using mineral standards. The EDS will have multi-

channel analyzer (MCA) calibrated for known peak energy centroids. Calibration will be make so as to have both low (1.0-3.0 KeV) and high (6.0-9.0 KeV) energy peaks fall within 0.05 KeV of its known centroid.

The magnification marker on the instrument will be checked once a week. This will be performed by following manufacturer instructions or by measurement of commercially available grids or licite spheres. Size measurements must be within 4 microns of certified values.

Phase identification procedures used in this SOP are based on semi-quantitative methods; therefore, daily standardization for all elements is not essential. Visual verification of element such as phosphorous or silica from an EDS spectra is sufficient. However, due to the spectral overlaps encountered by (Pb-S-Mo) and (As-Mg-Pb) and the difficulty in detecting oxygen and carbon it will be important to check their standardization routinely.

Initial calibration verification standards (ICVs) must be analyzed at the beginning of each analytical batch or once every 24 hours, whichever is more frequent. A set of mineral or glass standards will be run quantitatively for the metal of concern, sulfur, oxygen and carbon. If elemental quantities of the ICVs do not fall within +/- 5% of certified values for each element, the instrument must be recalibrated prior to analysis of investigative samples.

The metal-bearing forms in these samples will be identified using a combination of EDS, WDS and BEI. Once a particle is isolated with the backscatter detector, a 5-second EDS spectra is collected and peaks identified. The count rates for the metal(s) of concern, sulfur, carbon and oxygen can be either visually observed on the wavelength spectrometers or k-ratios calculated.

9.0 PERSONAL HEALTH AND SAFETY

Each individual operating the KEVEX x-ray fluorescence or electron microprobe instruments will have read the "Radiation Safety Handbook" prepared by the University (Quick Reference Guide and Table of Contents are supplied in Appendix A.) and follow all State guidelines for operation of x-ray equipment.

Latex gloves and particulate masks will be worn during preparation of sample cups. All material that comes in contact with the samples or used to clean work surface areas will be placed in poly-bags for disposal.

10.0 FINAL REPORT

A final laboratory report will be provided to the Contractor. The report will include all EMP data including summary tables and figures. Individual sample data will be provided on disk.

Speciation results will include: 1) a series of tables summarizing frequency of occurrence for each metal phase identified along with a confidence limit; 2) summary histograms of metal phases identified for each waste type; 3) a summary histogram of particle size distribution in each waste type; and 4) a summary of metal phase associations. Representative photomicrographs or TIFF images will also be included in the final report.

11.0 REFERENCES

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Table 2-1

Metal-Bearing Forms Found Within Western Mining Districts

OXIDES

- Lead Oxide
- Manganese lead oxide
- Iron lead oxide
- Lead molybdenum oxide
- Arsenic Oxide
- Cadmium Oxide
- Copper Oxide
- Zinc Oxide

SILICATES

- Slag
- Lead silicate
- Arsenic silicate
- Zinc silicate
- Clays

SULFATES

- Iron lead sulfate
- Lead sulfate
- Lead barite
- Zinc Sulfate
- Arsenic sulfate
- Copper sulfate

CARBONATES

- Lead Carbonate
- Zinc Carbonate

PHOSPHATES

- Lead phosphates

SULFIDES

- Lead sulfide
- Sulfur-containing salts
- Iron-arsenic sulfide
- Zinc sulfide
- Copper sulfides
- Copper-iron sulfide

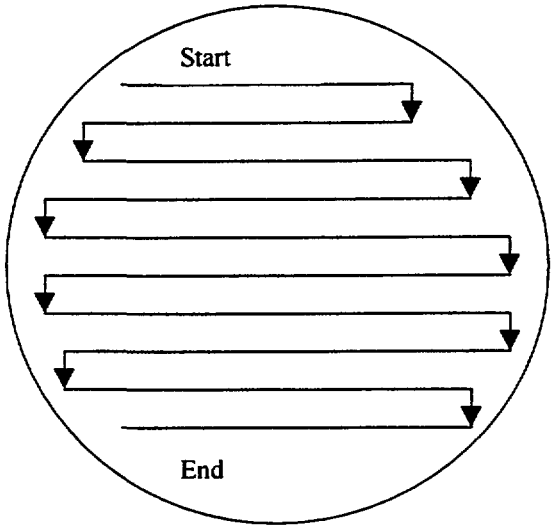
METALLIC

Lead, Zinc, Copper,Cadmium

- Lead/Arsenic/Cadmium Chlorides
- Lead paint
- Solder
- Organic lead
- Lead arsenate
- Lead vanadate

Minor telluride, and bismuth-lead phases

METALS SPECIATION



ASSAY _____ Pb ASSAY _____
SAMPLE ID _____ LAB _____
ANALYST _____ TIME END _____
TIME START _____

NOTES	IDENTITY	SIZE		Liberated	Inclusions	Incrusted Intergrown Cemented	Rim
		Length (um)	Area (um)				

Table 8-1

EMP Standard Operating Conditions

	WDS	EDS
Accelerating Voltage	15 KV	15-20 KV
Beam Size	1-2 microns	1-2 microns
Cup Current	10-30 NanoAmps	10-30 NanoAmps
Ev/Channel	NA	10 or 20
Stage Tilt	NA	Fixed
Working Distance	NA	Fixed
MCA time Constant	NA	7.5-12 microseconds
X-ray lines **	S K-alpha PET O K-alpha LDE1 C K-alpha LDEC Zn K-alpha PET As L-alpha TAP Cu K-alpha LIF Cd L-alpha PET Pb M-alpha PET Pb L-alpha LIF	S K-alpha 2.31 KeV O K-alpha 0.52 KeV C K-alpha 0.28 KeV Pb M-alpha 2.34 KeV Pb L-alpha 10.5 KeV Mo K-alpha 17.5 KeV Zn K-alpha 8.63 KeV Cu K-alpha 8.04 KeV As K-alpha 10.5 KeV As L-alpha 1.28 KeV Cd L-alpha 3.13 KeV

Table 8-2

Suggested Abbreviation for Photomicrographs

Metal-bearing Phase	Abbreviation
Lead Sulfide	Ga
Lead Sulfate	Ang
Lead Carbonate	Cer
Mn-(M) Oxide	Mn(M)
Fe-(M) Oxide	Fe(M)
(M)Phosphate	(M)Phoa
Fe-(M) Sulfate	Fe(M)Sul
Metal Oxide	(M)O
Pb-Mo Oxide	Wulf
Slag	Slag
Metallic Phase	(M)
Metal Silicate	(M)Si
Solder	Sold
Paint	Pnt
Metal-bearing Organic	(M)(Org)
(M) barite	(M)Bar
Pb arsenate	PbArs
Pb vanadate	PbVan
Greenockite	Gre
Chalcopyrite	Cp
Sphalerite	Sph
Arsenopyrite	Apy

APPENDIX B: Health-based Goals

TABLE 1: TOXICITY FACTORS & ASSOCIATED EFFECTS FOR CANCER AND NON-CANCER

Contaminant of Concern in Water	Source (NC/C)	Critical Effect	Non-cancer			Cancer		RBA	
			oRfD (mg/Kg-d)	Confidence	UF	Weight of Evidence	oSF (mg/Kg-d)	Soil	Dust
Metals									
Aluminum	d	Blood and liver effects	1.0E+00					1.0	1.0
Antimony	a	Longevity and blood effects	4.0E-04	Low	1000			1.0	1.0
Arsenic	a	Hyperpigmentation, keratosis	3.0E-04	Medium	3	A	1.5E+00	0.8	0.8
Barium	a	Hypertension	7.0E-02	Medium	3	D		1.0	1.0
Beryllium	a	Small intestinal lesions	2.0E-03	Low to Medium	300	B2		1.0	1.0
Cadmium	a	Human studies involving chronic exposures	1.0E-03	High	10	B1[e]		1.0	1.0
Chromium	a	No effects	5.0E-03	Low	500	A		1.0	1.0
Cobalt	b	Polycythencia	6.0E-02					1.0	1.0
Copper	f	Gastrointestinal irrritation	3.7E-02			D		1.0	1.0
Iron	d	Hemosiderosis	3.0E-01					1.0	1.0
Manganese	a	CNS effects	1.4E-01	Medium	1	D		1.0	1.0
Nickel	a	Decreased body and organ weights	2.0E-02	Medium	300			1.0	1.0
Selenium	a	Clinical selenosis	5.0E-03	High	3	D		1.0	1.0
Silver	a	Argyria	5.0E-03	Low	3	D		1.0	1.0
Thallium	a	Increased levels of SGOT and LDH	8.0E-05	Low	3000	D		1.0	1.0
Vanadium	a	Decreased hair cystine	9.0E-03		100			1.0	1.0
Zinc	a	Decrease in ESOD	3.0E-01	Medium	3	D		1.0	1.0
Mercury	c	Neurotoxicity	3.0E-04		30	D		1.0	1.0

NC/C - Non-cancer/Cancer
RBA - Relative Bioaccessiblity
a - IRIS Database
b - EPA Region III Database
c - HEAST
d - STSC
e - Weight of Evidence for inhalation only, not judged to be cancerous by oral pathway.
f - Based on a MCL of 1.3 mg/L (from HEAST); calculated by assuming an intake of 2 L/day by a 70 Kg human.

TABLE 2: RME EXPOSURE PARAMETERS

Parameter	Units	Population	
		Child	Adult
IR	mg/Kg-d	200	100
f_{soil}	--	0.45	0.45
f_{dust}	--	0.55	0.55
BW	Kg	15	70
EF	d/yr	350	350
ED	yr	6	24
AT - NC	d	2190	8760
AT - C	d	25550	25550
HIF - NC	Kg/Kg-d	1.3E-05	1.4E-06
HIF - C	Kg/Kg-d	2.7E-06	1.1E-06

NCI: Non Contact Intensive
IR: Intake Rate
BW: Body Weight
EF: Exposure Frequency
ED: Exposure Duration
HIF - NC: Human Intake Factor for Non-Cancer
HIF - C: Human Intake Factor for Cancer

TABLE 3: LEVEL OF HEALTH CONCERN FOR SOIL & DUST FOR A RESIDENT

LHCs IN SOIL AND DUST(mg/Kg)			
Chemicals of Concern in Soil or Sediment	HQ=1 Non-Cancer	RISK=1E-4 Cancer	Most Stringent
Metals			
Aluminum	9.2E+04	--	9.2E+04
Antimony	3.7E+01	--	3.7E+01
Arsenic	3.5E+01	3.5E+01	3.5E+01
Barium	6.5E+03	--	6.5E+03
Beryllium	1.8E+02	--	1.8E+02
Cadmium	9.2E+01	--	9.2E+01
Calcium	--	--	--
Chromium	4.6E+02	--	4.6E+02
Cobalt	5.5E+03	--	5.5E+03
Copper	3.4E+03	--	3.4E+03
Iron	2.8E+04	--	2.8E+04
Magnesium	--	--	--
Manganese	1.3E+04	--	1.3E+04
Nickel	1.8E+03	--	1.8E+03
Potassium	--	--	--
Selenium	4.6E+02	--	4.6E+02
Silver	4.6E+02	--	4.6E+02
Sodium	--	--	--
Thallium	7.4E+00	--	7.4E+00
Vanadium	8.3E+02	--	8.3E+02
Zinc	2.8E+04	--	2.8E+04
Mercury	2.8E+01	--	2.8E+01

HOUSEHOLD QUESTIONNAIRE

Name:

Address:

Telephone:

Neighborhood:

Date of Interview:

1. How many people live at this residence? _____

What are their names, sex, date of birth, and race?

#1. Name: _____Sex (M / F) DOB _____Race _____

#2. Name: _____Sex (M / F) DOB _____Race _____

#3. Name: _____ Sex (M / F) DOB _____Race _____

#4. Name: _____ Sex (M / F) DOB _____Race _____

#5. Name: _____ Sex (M / F) DOB _____Race _____

#6. Name: _____ Sex (M / F) DOB _____Race _____

* Fill out child questionnaire for each person born after 10/1/92

2. Is this residence a:

1) Trailer or mobile home
2) Single family home
3) Multiple family dwelling or apartment
4) Other _____

3. How many years has this family been living in this home?
(Interviewer- If this is a mobile home, check to see how long this family has lived in this mobile home at this location)

_____Years _____Months

If less than 3 months, obtain previous address:

Address: _____

4. What year was this house built? _____ 9999 = Don't know

5. Does your family have a vegetable garden on this property?
   1.) Yes
   2.) No

   If yes,

   5a) Do you can your vegetables?
   1.) Yes
   2.) No

   5b) What percentage of your vegetable intake consists of vegetables grown from your garden?

   _____%

6. *Interviewer:* Assess the Current Yard Condition:
   1.) Dirt or uncovered soil
   2.) Grass
   3.) An equal mix of grass and dirt
   4.) More dirt than grass
   5.) More grass than dirt
   6.) Gravel or stones
   7.) Asphalt or concrete
   8.) Other: _____

7. How many people in this household smoke tobacco inside the house? _____people

8. Which income level in the following list comes closest to the total gross household income (from all sources) for this family?

   1) Under 10,000 dollars
   2) Between 10,000 and 20,000 dollars
   3) Between 20,000 and 30,000 dollars
   4) Between 30,000 and 40,000 dollars
   5) Between 40,000 and 50,000 dollars

6) 50,000 dollars or more
7) Refused
8) Don't know

9. Has any member of the household worked at a smelter?

1.) Yes, Specify who and where: _____
Years of employment: _____

2.) No

10. Has any member of the household worked at the Rocky Mountain Arsenal?

1.) Yes, Specify who and where: _____
Years of employment: _____

2.) No

11. Does anyone in the household work with metals; for example, a battery plant worker, automotive mechanic, or welder?

1.) Yes, Specify who and where: _____
2.) No

12. Does anyone in your family play or spend time in the alley?

1.) Yes, Specify who, activity, and amount of time _____
2.) No
3.) Not Applicable

13.) Do you have any cats or dogs that go in and out of the house?

1.) Yes
2.) No

COMMENTS:

CHILD QUESTIONNAIRE

Name:

Address:

Telephone:

Neighborhood:

Date of Interview:

1. Child's Name: _____ Date of Birth _____

2. Child's Sex: Male Female

3. Child's Race:
   a.) White
   b.) Black
   c.) Hispanic or Latino or Chicano or Mexican
   d.) Asian
   e.) American Indian
   f.) Other (please specify _____)

4. How many years has the child lived at this residence?

   _____years

6. Where does <Child's Name> spend most of his/her daytime hours?
   a.) At home
   b.) At a babysitter
   c.) At a daycare center (specify _____)
   d.) At a relative's home (specify address _____)
   e.) Other (specify _____)

7. How many hours per day does <Child's Name> spend outside in yard?

   _____hours

8. How often does <Child's Name> take his/her bottle, pacifier, or food into the yard when he/she plays?

   a.) Always
   b.) Often
   c.) Sometimes
   d.) Rarely
   e.) Never

9. Many children put non-food items into their mouths. How often does <Child's Name> mouth the following:

ITEM	Always	Often	Sometimes	Rarely	Never
Toys					
Snow (in winter)					
Paper					
Dirt/sand/stones/leaves					
Cigarette butts					
Plaster					
Paint Chips					
Other					

COMMENTS: